

## Review

# Perspectives in Mixing-Assisted Oxidative Desulfurization of Fuel

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## Abstract

Oxidative desulfurization (ODS) is a promising alternative to conventional desulfurization methods because it operates under relatively mild conditions. Mixing-assisted oxidative desulfurization (MAOD), a variant of ODS, uses high-shear mixing to improve mass transfer between immiscible phases, enhancing sulfur conversion efficiency. This study reviews recent developments in MAOD for the removal of dibenzothiophene (DBT) and benzothiophene (BT) through a research matrix synthesizing the current literature. MAOD has demonstrated excellent performance, achieving up to 100% sulfur conversion in both model and real fuels, with DBT consistently showing higher conversion than BT. Response Surface Methodology (RSM) was commonly applied for process optimization, identifying typical operating conditions of 40–70 °C, 30 min reaction time, and 10,000 rpm as sufficient for complete sulfur conversion. The choice of oxidant and catalyst system strongly influences process efficiency, with polyoxometalates and hydrogen peroxide frequently achieving 100% conversion. Sustainable oxidants, including Fe(VI) and Mn(IV) derived from wastewater treatment sludge, have also shown promising results. However, the inability of model fuels to fully represent real fuel complexity and the limited use of RSM designs beyond Box–Behnken and Face-Centered Central Composite Designs highlight areas for future research. Overall, MAOD is a developing but highly promising desulfurization technology.

**Keywords:** alternative desulfurization method; benzothiophene removal; dibenzothiophene removal; high-shear mixing; mixing speed; oxidant–catalyst systems



Academic Editors: Fátima Mirante, Salete Balula and Gang Li

Received: 9 June 2026

Revised: 20 July 2026

Accepted: 27 July 2026

Published: 29 July 2026

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## 1. Introduction

Modern energy consumption and economic progress have been built on fossil fuels to sustain global energy demands since the Industrial Revolution. Compared to other sources, their high energy density [1] and widespread availability [2] make them ideal for powering industrial processes and supporting everyday activities. However, its sulfur content raises concerns for air quality and environmental sustainability as combustion produces substantial sulfur oxide (SO<sub>x</sub>) emissions [3]. This prompted the scientific community to explore ways to mitigate these emissions as industrialization continues to expand.

SO<sub>x</sub> forms when sulfur reacts with oxygen to produce sulfur dioxide (SO<sub>2</sub>). Photochemical reactions or contact with other oxidants in the atmosphere further oxidize it into sulfur trioxide (SO<sub>3</sub>). SO<sub>3</sub> can then react with water vapor to form sulfuric acid,

which serves as a precursor for acid rain, secondary aerosol, and other physicochemical atmospheric processes [4]. It also affects human health through promoting cardiovascular disease [5], respiratory issues [6], and type 2 diabetes [7]. Other environmental effects include damage to plantations [8], reduced soil respiration [9], and water acidification acceleration [10].

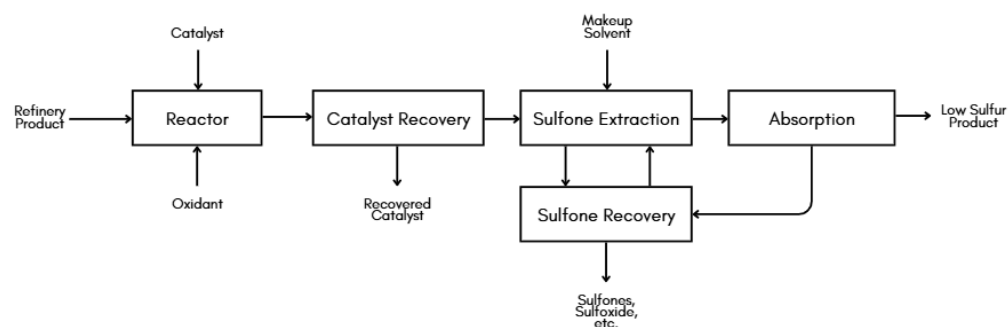
Regulations and policies have been adopted by government agencies to control sulfur emissions by setting a sulfur limit on fuel products. The United States regulates its sulfur by capping gasoline at a 10 ppm annual average and diesel at 15 ppm under its Tier 3 program [11]. China also implements strict sulfur limits with a maximum of 0.5% sulfur content for marine fuels within its Emission Control Areas. A maximum of 50 ppm sulfur content was set by the Philippine Department of Environment and Natural Resources [12]. The International Maritime Organization [13] also mandates maximum sulfur content of 0.50% (*w/w*) for marine fuels worldwide. In response to these regulatory pressures, industries are performing desulfurization technologies such as hydrodesulfurization (HDS) to lower the sulfur content of their fuel products.

HDS is a mainstream desulfurization process that involves the addition of hydrogen at high pressures and temperatures in the presence of a catalyst [14]. Despite its effectiveness, there is a significant financial burden to industries due to its high operating and construction costs. HDS requires a pressure of 3 MPa to 6 MPa and temperature of 300 °C to 400 °C to operate, which makes it energy-intensive [15]. It is also inefficient for deep desulfurization of aromatic sulfur compounds such as dibenzothiophene (DBT) and benzothiophene (BT) which are found in heavy oil fractions [16]. These serve as motivation for the exploration of other desulfurization strategies.

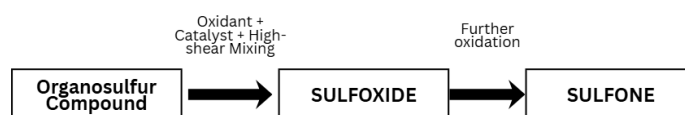
Oxidative desulfurization (ODS) is one of the promising alternative methods due to its high desulfurization efficiency without requiring hydrogen at lower temperatures and atmospheric pressure [17]. This would reduce operating costs in industries that are regulated in terms of sulfur emissions. ODS is a two-step process which involves oxidizing sulfur compounds into more polar sulfones and sulfoxides, followed by removal through extraction or thermal decomposition. ODS maximizes the conversion of organosulfur compounds into sulfoxides and sulfones using active oxygen species such as hydrogen peroxide ( $\text{H}_2\text{O}_2$ ). The resulting polar byproducts are then separated through extraction or adsorption. ODS can be combined with other techniques, such as ultrasound or photo-oxidation to increase efficiency [18]. The ODS mechanism involves three steps. The first step is the formation of active oxygen, where the oxidant interacts with the catalyst to form oxidizing agents. The use of  $\text{H}_2\text{O}_2$  forms  $\text{O}_2$  and  $\text{OH}^-$  active oxidant species. The catalyst is often composed of transition metals in the form of metal oxides [19], polyoxometalates [20], and supported catalysts [21]. Transition metals are typically utilized for their variable oxidation states that lowers activation energy and enables catalyst regeneration [22]. The attack of sulfur as the nucleophile on oxygen occurs as the second step. This forms sulfoxide out of the organosulfur. The final step is a second oxidation where the sulfoxide is further oxidized to form a sulfone [18]. The entire ODS process transforms non-polar sulfur compounds into more polar and heavier sulfones that can be removed easily through extraction and adsorption. Figure 1 illustrates the ODS process flow as derived from Said et al. [18].

Mixing-assisted oxidative desulfurization (MAOD) is a process-intensified variant of ODS that utilizes high-shear mixing to significantly enhance mass transfer between the organic and aqueous layers. By reducing droplet size and increasing interfacial areas, high-shear mixing facilitates more efficient contact between sulfur-containing compounds in the fuel phase and oxidizing species in the aqueous phase. More effective oxidation of sulfur compounds such as thiophenes and their derivatives consequently occurs. This

intensification minimizes mass transfer limitations, which are often the rate-controlling step in conventional ODS systems. Accelerated reaction kinetics and improving overall sulfur conversion efficiency are observed as a result. A generalized MAOD process flow is shown in Figure 2.



**Figure 1.** ODS process flow.



**Figure 2.** MAOD process flow.

MAOD is advantageous over HDS in terms of its thiophenic sulfur species removal, which can be sterically hindered at catalyst sites [23]. Limited reactivity persists due to restricted access to catalytic active sites and the requirement for elevated temperatures and pressures. MAOD addresses these limitations through liquid-phase oxidation rather than surface-catalyzed hydrogenation.

Numerous oxidant–catalyst systems have been explored for ODS, most commonly  $\text{H}_2\text{O}_2$  with phosphotungstic acid (HPW). This system is widely studied due to their strong oxidative capability and relative operational simplicity. There is also the emergence of heterogeneous and green catalysts to promote better recoverability and reusability potential [24]. This promotes the sustainability and industrial feasibility of the process.

Although MAOD has attracted increasing attention in recent years, research in this field remains relatively limited due to its recent emergence. Existing studies focus on key process parameters, such as mixing speed, mixing time, catalyst dosage, oxidant concentration, and reaction temperature, on MAOD efficiency. There is a significant research gap due to the lack of comprehensive review articles that systematically evaluate the diverse oxidant–catalyst systems utilized in MAOD processes. The novelty of this review is to comprehensively collate on the existing and recent MAOD-related developments in oxidant and catalyst systems. It examines parametric trends, performance outcomes, and identified limitations across studies to highlight potential directions for future research.

This study aims to summarize key process parameters for optimal sulfur conversion and highlight research gaps to guide future efforts toward more sustainable and cost-effective MAOD processes. This review focuses on MAOD studies indexed in Scopus from 2014 to the present, specifically addressing thiophenic sulfur compounds. It considers only the initial step of the ODS reaction, namely the oxidation of sulfur compounds to their corresponding sulfones. Section 2 elaborates on the collection and review strategies for the collected journal articles. Section 3 details the results of the review process and highlights the noted research gaps. The main findings are summarized and concluded in Section 4.

## 2. Methodology

### 2.1. Collection and Review of Related Journal Articles

The literature review was conducted by collating research articles that fit the selection and exclusion criteria of the review paper. The timeframe includes only research articles from 2014 to 2025. The database that was used in the review is the Scopus database and only articles indexed in Scopus is included. The review does not include other review articles and conference papers. Boolean Operators were used as a search strategy using keywords such as “mixing-assisted” and “high-shear mixing”. Research articles that apply high-shear mixing as process intensification of oxidative desulfurization in the removal of sulfur compounds from model and/or real fuel oil were gathered. Inclusion was limited to articles that show a maximum percent sulfur conversion and indicate the target sulfur compounds. A wildcard operator (\*) to return articles containing any form of the root word “desulfur” was used. Keywords such as “mixing-assisted” and “high-shear mixing” was included using AND and OR operators. The search was also limited to journal articles using a document specifier. The specific search string used in Scopus is as follows: ((“oxidative desulfur\*”) AND (“mixing-assisted” OR “high-shear mixing”)) AND (LIMIT-TO (DOCTYPE, “ar”)).

### 2.2. Evaluation of Journal Articles to Be Included in the Review Paper

The use of a thematic research matrix was employed to systematically evaluate and synthesize the collected research articles to be included in the review paper. A structured table was created listing the study title, authors and year of publication. The parameters on review specifically include the target sulfur compounds and the type of fuel used. The oxidant/catalyst system used and the operating parameters that exhibit the highest sulfur conversion or removal have been reviewed to create the matrix.

### 2.3. Identification of Research Gap

Research gaps and areas of potential future studies have been identified through synthesis of the research matrix. The collection of studies focused on converting DBT and BT into dibenzothiophene sulfone (DBTO<sub>2</sub>) and benzo thiophene sulfone (BTO<sub>2</sub>) in either model fuel or real fuel. However, real fuels contain more than these two compounds, whose conversion remains unstudied. Most of the studies utilize a common oxidant–catalyst system using POMs and H<sub>2</sub>O<sub>2</sub>. While these conventional systems achieve high conversion values up to 100%, the possibility of more sustainable sources of reagents such as treatment processes wastes which bridge circular economy is left uninvestigated. The systems in the studies are also limited to lab-scale treatment processes since mixing-assisted oxidative desulfurization is still a relatively new technology. Feasibility studies regarding its applicability for industrial scale systems are a possible area for future studies. The research gaps identified through the evaluation of research matrix were further discussed in the following sections.

## 3. Research Matrix

The research matrix in Table 1 shows how different oxidant/catalyst systems perform on converting the target sulfur compounds to their respective sulfones. The optimal operating parameters that achieved the highest sulfur conversion are also listed for systems that use model fuel only. MAOD can achieve a high amount of percent sulfur conversion up to 100% conversion using a typical H<sub>2</sub>O<sub>2</sub> and HPW system [25]. Some studies utilizing other oxidant–catalyst systems from more sustainable sources such as crude Fe(VI) and Mn(IV) derived from water treatment sludge succeed in achieving relatively high percent conversion values as well [26].

**Table 1.** Existing studies on MAOD systems using model fuel.

| Target Sulfur Compounds | Oxidant/Catalyst System                                                            | Operating Parameters                                                                                                                                                          | Percent Sulfur Conversion    | Reference           |
|-------------------------|------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------|---------------------|
| DBT                     | Oxidant: Fe (VI) and Mn (IV)                                                       | Temperature: 60 °C<br>Operating Time: 30 min<br>Mixing Speed: 5000 rpm<br>Oxidant-DBT ratio: 9:1<br>Oxidant-PTA ratio: 1:1                                                    | 100%                         | Chen et al. [26]    |
| BT                      | Oxidant: Crude Fe (VI)                                                             | Temperature: 40 °C<br>Operating Time: 30 min<br>Mixing Speed: 10,800 rpm<br>Oxidant Concentration: 600 ppm<br>PTA Concentration: 101 mg/50 mL                                 | 88.1%                        | Haboc et al. [23]   |
| DBT                     | Oxidant: Crude Fe (VI)                                                             | Temperature: 60 °C<br>Operating Time: 30 min<br>Mixing Speed: 4400 rpm<br>Oxidant Concentration: 500 ppm<br>PTA Concentration: 200 mg/50 mL                                   | 99.6%                        | Haboc et al. [27]   |
| DBT and BT              | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: Activated-Carbon supported HPW | Temperature: 65 °C<br>Operating Time: 90 min<br>Mixing Speed: 16,800 rpm                                                                                                      | 61.73%                       | Barilla et al. [28] |
| DBT                     | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: HPW                            | Temperature: 50 °C<br>Operating Time: 3–50 min<br>Mixing Speed: 5000 rpm                                                                                                      | 100%                         | Wan et al. [25]     |
| DBT                     | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: HPW                            | Temperature: 40 °C<br>Operating Time: 5–30 min<br>Mixing Speed: 10,000 rpm<br>PTA-Oxidant ratio: 1:1                                                                          | 100%                         | De Luna et al. [29] |
| DBT and BT              | Oxidant: Fe (VI)                                                                   | DBT:<br>Temperature: 51.26 °C<br>Operating Time: 14.43 min<br>Mixing Speed: 8704 rpm<br>BT:<br>Temperature: 52.22 °C<br>Operating Time: 15.42 min<br>Mixing Speed: 12,198 rpm | 93.68% (DBT),<br>84.35% (BT) | Choi et al. [30]    |
| DBT and BT              | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: Polyoxometalates               | Temperature: 30–70 °C<br>Operating Time: 2–30 min<br>Mixing Speed: 10,000 rpm                                                                                                 | 100%                         | Choi et al. [31]    |
| DBT and BT              | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: Polyoxometalates               | Temperature: 70 °C<br>Operating Time: 30 min<br>Mixing Speed: 10,000 rpm                                                                                                      | 97.4%                        | Choi et al. [32]    |
| DBT and BT              | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: HPW                            | Temperature: 70 °C<br>Operating Time: 30 min<br>Mixing Speed: 10,000 rpm                                                                                                      | 97%                          | Lu et al. [33]      |

Sulfur conversion refers to the oxidation of sulfur-containing compounds into their corresponding sulfoxides and sulfones. Sulfur removal refers to the reduction of sulfur content in the fuel following oxidation and subsequent separation of the oxidized sulfur species.

Table 2 shows the research matrix that lists the oxidant/catalyst systems currently used to treat real fuel. The optimal operating parameters that achieved the highest sulfur conversion are listed along with the fuel type used. Systems using model fuel commonly employ H<sub>2</sub>O<sub>2</sub> with POM catalyst systems, particularly tungsten-based POMs. MAOD

systems can achieve 100% sulfur conversion for raw diesel [33]. Higher sulfur conversion can be observed for systems using raw diesel than other fuel systems. In a comparative study by De Luna et al. [34] it is observed that CSTR is a better mixer than an in-line mixer. The sulfur conversion for systems using pyrolysis oil is noticeably low in terms of the conversion values that are listed in the matrix. It is worth noting that both the systems that tested pyrolysis oil used an oxidant-only system with crude Fe(VI) as the catalyst, as shown by Haboc et al. [23,27]. MAOD systems are also shown to be effective in treating sour gas using acids and nitrogen dioxide as the oxidants achieving a 95% sulfur conversion [35].

**Table 2.** Existing studies on MAOD systems on real fuel.

| Fuel Type           | Oxidant/Catalyst System                                                      | Operating Parameters                                                                                                                                                                                                                            | Percent Sulfur Conversion         | Reference           |
|---------------------|------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------|---------------------|
| Pyrolysis Oil       | Oxidant: Crude Fe (VI)                                                       | Temperature: 40 °C<br>Operating Time: 30 min<br>Mixing Speed: 10,800 rpm<br>Oxidant Concentration: 600 ppm<br>PTA Concentration: 101 mg/50 mL                                                                                                   | 54.7%                             | Haboc et al. [23]   |
| Pyrolysis Oil       | Oxidant: Crude Fe (VI)                                                       | Temperature: 41.7 °C<br>Operating Time: 30 min<br>Mixing Speed: 8157 rpm<br>Oxidant Concentration: 537 ppm<br>PTA Concentration: 114 mg/50 mL                                                                                                   | 53.2%                             | Haboc et al. [27]   |
| Raw Diesel          | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: NaPW                     | Temperature: 62.8 °C<br>Operating Time: 29.6 min<br>Mixing Speed: 8800 rpm                                                                                                                                                                      | 96.6%                             | Choi et al. [36]    |
| Sour Gas Condensate | Oxidant: H <sub>2</sub> SO <sub>4</sub> , HNO <sub>3</sub> , NO <sub>2</sub> | Temperature: 50 °C<br>Operating Time: 30 min<br>Mixing Speed: 5000 rpm                                                                                                                                                                          | 95.61%                            | Pouladi et al. [35] |
| Raw Diesel          | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: HPW                      | CSTR: Temperature: 70 °C<br>Reaction Time: 0.8–4 min<br>Diesel Flowrate: 200 mL/min<br>Mixing Speed: 18,000 rpm<br>In-line Mixer:<br>Temperature: 70 °C<br>Residence Time: 0.3–6 min<br>Diesel Flowrate: 200 mL/min<br>Mixing Speed: 18,000 rpm | 98% (CSTR)<br>93% (In-line Mixer) | De Luna et al. [34] |
| Raw Diesel          | Oxidant: H <sub>2</sub> O <sub>2</sub><br>Catalyst: HPW                      | Temperature: 70 °C<br>Operating Time: 30 min<br>Mixing Speed: 10,000 rpm                                                                                                                                                                        | 100%                              | Lu et al. [33]      |

### 3.1. Parametric Trends in the MAOD Experimental Approach

All reviewed studies share similarities in their experimental approaches in delving into MAOD. DBT and BT as aromatic heterocycles are predominantly used as target sulfur compounds because of their resistance to conventional HDS techniques. This allows the simulation of hard-to-remove sulfur, which allows proper assessment of the effectiveness of alternative desulfurization techniques. Isolating DBT or BT as the model sulfur compound allows the comparison of catalyst performance with minimal interference arising from complex mixtures. The studies on MAOD are rooted in the general objective of lessening sulfur compounds in fuel mixtures to prevent harmful release of sulfur oxides. As such, all studies are concerned on which parameters affect the efficiency of the MAOD system. To provide a perspective on how effective a MAOD system can be applied, the review discussed trends regarding these operational parameters. The discussion showed where the current research on MAOD agrees and its progress towards real life applications.

### 3.1.1. Oxidant/Catalyst System

Discussion of the trend on the catalyst systems used in MAOD systems is shown in this section. For most of the studies, a convention of using POMs and  $\text{H}_2\text{O}_2$  as the system is observed. HPW/PW is the most common catalyst as seen in the studies of De Luna et al. [34], Lu et al. [33], and Wan et al. [25]. DBT and BT have an electron density of 5.758 and 5.739 respectively [37], indicating high susceptibility to electrophilic oxidation at the sulfur atom.  $\text{H}_2\text{O}_2$  is therefore an effective oxidant as it preferentially reacts with sulfur species within an electron density range of approximately 5.716 to 5.739 under mild conditions. The adoption of POMs as a catalyst stems from the reaction of the transition metal derivatives in the POMs which react with  $\text{H}_2\text{O}_2$  to form peroxocomplexes [38]. Peroxocomplexes are stronger and more selective than their parent peroxides and are effective for both inorganic and organic substrates [39]. The use of oxidant-only systems has been explored using Ferrate. One out of four studies used commercial Fe(VI) [30]. The other three used Fe(VI) derived from drinking water treatment plants because of the cost and availability limitations of commercial Fe(VI). Sludge-derived Fe(VI) as an oxidant is studied by Haboc et al. [23,27]. The use of sludge-derived Mn(VI) and Fe(VI) has been investigated by Chen et al. [26,40]. All four studies utilized Fe(VI) in the form of potassium ferrate ( $\text{K}_2\text{FeO}_4$ ) in acidic media. Fe(VI) is employed as an oxidant due to its high standard reduction of 2.2 V in acidic media [41], stability and selectivity [42], and ability to self-decompose into non-toxic products [43]. Moreover, using sludge-derived Fe(VI) and Mn(VI) bridges the gap of cost and sustainability. Iron-rich compounds are commonly used in sludge treatment [44]. Fe(VI) can be synthesized through various chemical and electrochemical methods [45].

### 3.1.2. Temperature

Temperature ranged from 40 to 70 °C across most studies. Higher temperatures generally favor greater sulfur conversion as supported by studies of Barilla et al. [28] and Choi et al. [31]. This trend can be attributed to the temperature dependence of reaction kinetics, where higher temperatures increase the reaction rate constant according to the Arrhenius relationship [46]. At higher temperatures, the formation of metallic peroxide increases [47]. The acceleration of the oxidation of sulfur-containing compounds occurs as a result. A favorable mass transfer between the oil and oxidant also takes place at higher temperatures. This facilitates more effective contact between the sulfur compounds and oxidizing agents. Excessively high temperatures may reduce the removal efficiency. This is caused by the decrease in oxidant concentration via the decomposition of oxidizing agents such as  $\text{H}_2\text{O}_2$  [48]. Higher temperature also promotes the evaporation of volatile compounds [49].

Elevated temperatures have also promoted enhanced emulsification between the oil and oxidant phases by reducing interfacial tension and lowering viscosity [50]. The decrease in viscosity aids in the formation of emulsion bubbles [51]. Through increasing the interfacial area available for reaction, more frequent contact between sulfur-containing compounds and oxidizing species occurs [52]. This further contributes to the observed increase in desulfurization efficiency.

### 3.1.3. Mixing Speeds

Mixing speed affects mass transfer between phases in ODS. Mixing speeds were tested in a range from as low as 5000 rpm [26] to up to 18,000 rpm [34]. Many studies adopted 10,000 rpm as the baseline, as seen in the works of De Luna et al. [29] and Choi et al. [31]. An increase in mixing speed enhances turbulence and promotes reactant contact by reducing boundary layer thickness around droplets and improving the dispersion

of reactants [53]. The increase in contact frequency between reactants increases the reaction rate. This explains the observed direct relationship between agitation speed and desulfurization efficiency.

However, Barilla et al. [28] has noted that ANOVA results do not support the significance of mixing speed in sulfur oxidation response. This occurs when the reaction is already limited by chemical kinetics rather than mass transfer. The molecules may already be in contact, but the chemical reaction itself is slow. An increase in mixing speed would not make a difference in the circumstances due to the bulkiness of the compound or the lack of reactivity in the oxidizing agent. Plateauing of the increase in efficiency at greater mixing speeds can also be experienced because of the back mixing of the smaller droplets due to turbulence [54].

Choi et al. [30] also reveals that optimum speed varies with the model compound. The determined optimal agitation speeds were 12,198 rpm for BT and 8704 rpm for DBT. This may be attributed to the higher reactivity of DBT, which lessens the agitation necessary for effective interaction with the oxidizing agent.

#### 3.1.4. Reaction Time

The reaction time refers to the amount of time the sulfur compounds and oxidizing agents spent inside a batch process system under the operating conditions. Residence time is used for continuous flow systems. The review across different studies shows that the reaction time affects sulfur conversion, even if it interacts with mixing intensity, catalyst system, and fuel complexity. The reaction time used by the studies ranged around 14 min to 30 min, but some studies explored reaction times of 3 min [36] to 90 min [28].

The most common optimal reaction time across the studies is 30 min as applied by Pouladi et al. [35] and Chen et al. [26], suggesting that this time is sufficient for sulfur oxidation. The conversion for these studies remained above 94%. The sulfur conversion was 93.68% at 14.43 min and 84.35% at 15.42 min for DBT and BT in studies that utilized shorter reaction time [35]. At 90 min reaction time, the sulfur conversion only reached 61.73% [28]. This suggests that an increased reaction time does not guarantee higher sulfur conversion due to catalyst limitations, oxidant degradation, decrease in selectivity, different reactor configurations, and mass transfer limitations.

The pseudo-first-order kinetics which are followed by the MAOD oxidation reactions also explain the diminishing returns of longer reaction times [34]. When a large excess of one reaction, when compared to the other, barely changes in concentration during the reaction, the reaction behaves as a first-order reaction with respect to the other reaction. MAOD employs oxidants in large excess. This makes the reaction more dependent on the sulfur compound. By the 30 min mark, the system approaches near-complete conversion as rapid conversion occurs early in the reaction.

#### 3.1.5. Optimization Strategies

The studies that adopted optimization as part of its methodology generally relied on Box–Behnken Design (BBD) as its Response Surface Methodology (RSM), as studied by Choi et al. [30] and De Luna et al. [34]. RSM involves designed experiments to map the relationship between multiple variables and predict optimal conditions. BBD is applied as an independent quadratic design that determines possible treatment conditions, such as the midpoints of edges and center of a process space. This is effective in studying interaction effects [55]. Barilla et al. [28] explored the Face-Centered Central Composite (FCC) Design as its RSM. This involves placing axial points at the center of each face of the experimental cube, using the low, middle, and high levels. Factorial corner points are considered, so extreme combinations are tested. The wide adoption of RSM suggests that MAOD systems

are non-linear processes. The existence of curves makes it so that simple linear fitting is not sufficient and quadratic terms need to be used to determine the optimal range [56].

### 3.2. Performance Patterns on Sulfur Conversion

Studies consistently achieve an above 85% desulfurization efficiency rate when model sulfur is used. The work of Choi et al. [30] stands as an exception with its 84.35% desulfurization efficiency when BT was utilized. Haboc et al. [23] also achieved a lower desulfurization rate of 78.5%, with BT as the model sulfur compound. This supported the earlier claim that DBT has a better sulfur reduction capacity than BT [35].

Electron density and structural differences explain the higher reduction capacity of DBT. The higher electron density in DBT compared to BT eases the attack of the oxidizing agent, which acts as an electrophile [57]. The sulfur in DBT is divalent, which means it is bonded to two carbon atoms in aromatic rings. The sulfur has an oxidation state of +2. The oxygen atoms are electrophilically added to this divalent sulfur, which converts it stepwise to sulfoxide, then to a hexavalent sulfone.

DBT and BT are both heteroaromatic compounds with sulfur inside a thiophene ring. BT is benzene and thiophene fused together to form one bicyclic molecule. DBT is structured with a thiophene ring in the middle, with two benzene rings fused at the sides. This makes DBT tricyclic. The extra benzene ring in DBT increases the electron density around the sulfur atom through resonance. This makes DBT more prone to electrophilic oxidation [58].

A decrease in efficiency is generally observed when real fuels are substituted to undergo the proposed desulfurization processes due to their intricate composition, compared to model fuels. For example, Haboc et al. [27] observed a drop from 99.6% to 53.2% desulfurization efficiency when pyrolysis oil was adopted as the test sample instead of DBT. Real fuels contain compounds with greater steric hindrance, aromatic structures, and experience additional phase-transfer limitations due to their bulkiness. Real fuels that were utilized include diesel oil and pyrolysis oil as studied by Choi et al. [30] and Choi et al. [36].

### 3.3. Comparison with Other Assistive-Oxidative Desulfurization Methods

There are several other assistive-oxidative desulfurization methods that have been explored by studies. These generally involve the use of emerging technologies designed to enhance sulfur conversion from liquid fuels. Most techniques remain at the laboratory stage, with ultrasound-assisted ODS being the most developed approach.

Boshagh et al. [59] reviewed eight major assistive ODS techniques, namely ultrasound-assisted, plasma, microwave-assisted, hydrodynamic cavitation-assisted, photocatalytic, electrochemical, magnetic field-assisted, and bio-oxidative desulfurization. The mixing-assisted oxidative desulfurization technique only emerged recently.

The ultrasound-assisted oxidative desulfurization (UAOD) utilizes ultrasound probes to magnify the physical and mechanical effects of the desulfurization process [60]. The high-frequency sound waves generate micro-bubbles in the liquid, generating localized spots with extreme conditions. Smoother dispersion capabilities and increased mass transfer and reaction kinetics were observed in heterogeneous reaction systems as a result [61]. Compared to MAOD, UAOD has shown to exhibit higher desulfurization efficiency within shorter reaction times. However, equipment costs may be higher or more specialized, which makes scalability challenging. MAOD is still identified for future research and development [18].

Plasma oxidative desulfurization utilizes non-thermal plasma to generate highly reactive oxygen species, such as ozone, atomic oxygen, and hydroxyl radicals [62]. Electrical discharge allows the interaction between energetic electrons and oxygen-containing gases that attack electron-rich sulfur atoms in organosulfur. Sulfoxides, and ultimately sulfones,

are formed, which can be efficiently separated due to their increased polarity [63]. The high efficiency of this method is largely attributed to the non-equilibrium nature of plasma, where electrons possess significantly higher energy than the surrounding gas molecules. This allows rapid generation of reactive species and facilitates fast oxidation kinetics, even under ambient conditions [64]. Plasma oxidative desulfurization demonstrates high efficiency under ambient conditions, with reaction times on the order of seconds to minutes as compared to MAOD. However, this approach is associated with greater process complexity due to its need for high-voltage power supply [65], plasma reactor [66], and electrode-dielectric materials [67].

Microwave-assisted ODS maximized electromagnetic radiation for the generation of volumetric heat for the enhancement of reaction kinetics [68]. Kaeed et al. [69] reveals that this technique may achieve >97% sulfur conversion using 800 W and 10 min parameters, with the advantage of rapid heating and uniform temperature distribution. While MAOD relies on the mechanical enhancement of mass transfer, microwave-assisted ODS have shorter optimal reaction times of 10 min when compared to the 30 min reaction time of UAOD.

Hydrodynamic cavitation-assisted oxidative desulfurization (HCAOD) relies on the generation of intense, localized heat and pressure through liquid pressure changes to facilitate faster reaction kinetics and shorter processing times [70]. It breaks down sulfur-containing compounds using an oxidant and catalysts, such as  $\text{H}_2\text{O}_2$  and formic acid respectively, at mild operating temperatures [71]. While studies on MAOD efficiency on real fuel remains limited, Baradaran and Sadeghi [72] have shown that 95% extent of desulfurization can be achieved for the treatment of diesel fuel feedstock. This study adopted optimum conditions with a HC pressure drop of 4.2 bar and acid to oxidant ratio ( $n_A/n_O$ ) of 3.2 at 29 min. However, HCAOD may present greater operational complexity than MAOD due to the need for precise control of pressure drop and cavitation conditions. MAOD systems are generally simpler and more straightforward to scale. This makes it a more favorable option for industrial applications despite potentially longer reaction times.

Photocatalytic oxidative desulfurization (PODS) employs semiconductor-based catalysts that are activated by light irradiation to oxidize sulfur-containing compounds [73]. The formed radical species in the form of hydroxyl and superoxide radicals facilitate the oxidation of organosulfur compounds to be extracted from the fuel matrix. This process is initiated by the excitation of the photocatalyst which generates electron-hole pairs that participate in redox reactions [74]. The continuous production of reactive oxygen species occurs as a result. The efficiency of PODS is further enhanced through the development of heterojunction photocatalysts. This improves charge separation and extends light absorption into the visible region to increase photocatalytic activity [75]. Zhao et al. [76] has shown that approximately 98–99% DBT conversion rate can be achieved at a five-cycle recyclability feature. Compared to MAOD, PODS reduces reliance on mechanical mixing by utilizing light as the primary energy input and demonstrates strong potential for oxidizing refractory sulfur compounds. However, MAOD remains more practical in terms of scalability and operational simplicity. PODS is limited by photocarrier recombination, restricted light utilization, and catalyst stability issues, with most applications still at the laboratory scale [77].

Electrochemical-assisted oxidative desulfurization (EAODS) is a variant of assisted-oxidative desulfurization that leverages electrochemical reactions to generate in situ oxidizing species [78]. While MAOD relies on the enhancement of mass transfer mechanisms through mixing, EAODS shifts the process mechanism by producing reactive oxidants directly at the electrode surface. Sulfur-containing compounds are oxidized by electrochemically generated species such as active oxygen and oxidants like hypochlorite ( $\text{ClO}^-$ ),

formed at the anode [79]. These species facilitate the oxidation of organosulfur compounds into sulfoxides and sulfones, which can then be readily separated. Tang et al. [80] showed that a 99.62% desulfurization efficiency in condensate gasoline is possible, while Cao et al. [81] demonstrated 100% sulfur conversion within 20 min without external oxidants or catalysts. EAODS offers relatively mild operating conditions, minimal waste generation, and electrode tunability compared to conventional HDS [82]. While MAOD relies on externally supplied oxidants and catalysts, EAODS generates reactive species in situ, which reduces chemical demand. MAOD has demonstrated potential for industrial implementation due to its ability to achieve high sulfur conversion rates. However, further scale-up studies are required to fully evaluate its industrial feasibility. EAODS faces challenges related to electrode stability, energy consumption, reactor design, and the scale-up of electrochemical systems [83].

Jin et al. [84] revealed that a magnetic field-assisted oxidative desulfurization (MFA-ODS) process improves sulfur conversion through intensified mass transfer. The facilitated recovery of magnetics absorbance and localized heating effects allows 98% efficiency within 30 min at 70 °C. It adopts magnetic fields to improve mass transfer, reaction collision frequency, and catalytic activity at lower temperatures when compared to conventional methods. MFA-ODS reduces reliance on mechanical agitation by employing an external magnetic field [85]. However, the simplicity and ease of implementation of MAOD remains favorable.

Bio-oxidative desulfurization (BODS) is explored due to the environmental advantages it offers, despite the practical limitations it faces for industrial deployment. This process adopts microorganisms at ambient temperatures and pressures to convert sulfur compounds through enzymatic pathways. Hydrogen and metal catalysts are not required [86]. BODS has shown excellent selectivity with reduced energy costs, low carbon emissions, and a lack of unfavorable byproducts. Slow reaction rates remain a pressing challenge with BODS [87]. The efficiency level reached is highly dependent on the microorganisms and conditions used. The use of the *Rhodococcus* strain has shown to have an efficiency of 80–90%. This is lower than the efficiency achieved by MAOD of over 90% [88].

To illustrate the difference in performance trends of the various assistive oxidative desulfurization techniques, a comparative table was made. Optimal reaction time and temperature were considered as the two standard parameters. The various techniques greatly differ in operation, which makes comparison regarding parameters hard. A respective operating parameter column was made to give context for each assistive technique. The comparisons are tabulated in Table 3.

The reported performance metrics were obtained from different studies employing varying sulfur model compounds, fuel matrices, catalysts, oxidants, reactor configurations, and operating conditions. Direct quantitative comparison among the assistive oxidative desulfurization techniques should be interpreted with caution. The values are provided to illustrate general performance trends and reported operating ranges rather than to establish definitive rankings among the technologies. Certain metrics, such as energy consumption, cost, and technology readiness level, were not consistently reported across the literature and therefore could not be comprehensively compared.

**Table 3.** Comparison between assistive oxidative desulfurization techniques.

| Assistive Technique    | Oxidant/Catalyst System                                | Maximum Sulfur Conversion | Optimal Temperature | Optimal Reaction Time | Representative Operating Conditions                        | Reference |
|------------------------|--------------------------------------------------------|---------------------------|---------------------|-----------------------|------------------------------------------------------------|-----------|
| MAOD                   | H <sub>2</sub> O <sub>2</sub> /HPW                     | 100%                      | 70 °C               | 30 min                | Mixing speed: 10,000 rpm                                   | [31]      |
|                        | H <sub>2</sub> O <sub>2</sub> /NaPW                    | 97.4%                     | 70 °C               | 30 min                | Mixing speed: 10,000 rpm                                   | [32]      |
| UAOD                   | H <sub>2</sub> O <sub>2</sub> /NaPW                    | 94.8%                     | 70 °C               | 30 min                | Frequency: 20 kHz<br>Power: 200 W                          | [32]      |
| Plasma ODS             | DBD Plasma                                             | 93.78%                    | 50 °C               | 240 min               | Voltage: 11 kV<br>Air flow: 75 mL/min                      | [89]      |
|                        | DBD Plasma /FeCl <sub>3</sub> -SiO <sub>2</sub>        | 98.4%                     | 25 °C               | 30 min                | Voltage: 30 kV<br>Air flow: 90 mL/min<br>Frequency: 600 Hz | [62]      |
| Microwave-assisted ODS | H <sub>2</sub> O <sub>2</sub> /vanadyl acetylacetonate | 86.67%                    | 80 °C               | 1.5 min               | Power: 500 W                                               | [90]      |
|                        | H <sub>2</sub> O <sub>2</sub> /CuO-ZnO nanocomposite   | 93%                       | Not specified       | 15 min                | Power: 540 W<br>Catalyst dosage: 8 g/L                     | [91]      |
| HCAOD                  | H <sub>2</sub> O <sub>2</sub> /Formic acid             | 95.8%                     | 50 °C               | 30 min                | Pressure drop: 5 bar                                       | [92]      |
| PODS                   | H <sub>2</sub> O <sub>2</sub> /Ag@AgBr/Al-SBA-15       | 98.66%                    | 70 °C               | 360 min               | Catalyst dosage: 0.05 g                                    | [93]      |
|                        | H <sub>2</sub> O <sub>2</sub> /Perovskite composites   | 99%                       | 80 °C               | 60 min                | Power: 18 W<br>Catalyst dosage: 0.1 g                      | [76]      |
| MFA-ODS                | H <sub>2</sub> O <sub>2</sub> /Heteropolytungstate     | 98%                       | 70 °C               | 30 min                | Magnetic field: 17.3 mT<br>Catalyst dosage: 5%             | [84]      |
| EAODS                  | In situ generation using Graphite electrodes           | 92.67%                    | 50 °C               | 60 min                | Cell voltage: 4 V<br>Mixing speed: 500 rpm                 | [78]      |
| BODS                   | Desulfurization using microorganisms                   | 96.4%                     | 30 °C               | 4200 min              | Strain: <i>Rhodococcus</i> spp. (SL-9)                     | [94]      |

### 3.4. Research Gaps and Future Directions

The review of the included MAOD studies reveals several research gaps. This has been organized into thematic subsections, namely model fuel versus real fuel complexity, limitations of current optimization methodologies, catalyst development and sustainability considerations, scale-up and techno-economic considerations, and limitations on MAOD.

#### 3.4.1. Model Fuels Versus Real Fuel Complexity

The constraint of model sulfur compounds on DBT and BT highlights opportunities to explore DBT-BT mixtures to more accurately replicate the composition of real fuels. Alkylated DBT, such as 4,6-dimethyldibenzothiophene, also remains underexplored. It may provide insight into the oxidation efficiency of ODS in more difficult-to-treat fuels. Alkylated DBT is harder to oxidize due to hindrance from methyl groups.

Another gap lies in the reliance on model fuels. The behavior of actual fuels is not fully captured. They do not fully capture the behavior of actual fuels. Actual fuels are composed of hydrocarbon mixtures of paraffins, naphthene, aromatics, and other sulfur compounds. Studies using real fuel in the form of diesel and pyrolysis-derived fuels with varying octane numbers are limited. This introduces difficulty in understanding differences in oxidation and desulfurization performance between model and practical systems. Comparative studies are also lacking between model fuels, pyrolysis fuels, and commercial diesel. Valuable insight on the response of different fuel types to oxidation and desulfurization processes under similar conditions would be provided.

#### 3.4.2. Current Optimization Methodologies

Optimization methods among the papers are also limited to RSM, specifically BDD and FCC. This leaves opportunities for exploration of other methodologies, such as the Central Composite Design (CCD) and D-Optimal design. CCD considers axial points

beyond the factorial range, which have been applied in other optimization studies [95]. D-Optimal design is based on a computer-generated design for irregular factor ranges. As optimization tasks become adjustable, specific portions of the response surface can be focused on [96].

#### 3.4.3. Catalyst Development and Sustainability Considerations

There is also opportunity to explore the application of MAOD using other eco-friendly catalysts. Zhang et al. [97] synthesized  $\text{UiO-66}(\text{Zr}/\text{Sn}^{2+})$  catalysts using solvent-free methods and achieved 100% DBT removal with  $\text{H}_2\text{O}_2$  and excellent reusability over multiply cycles. HKUST-1 is also a candidate catalyst for MAOD due to its strong affinity toward sulfur-containing compounds from its porous structure and accessible Cu active sites [98].

#### 3.4.4. Scale-Up and Techno-Economic Considerations

The literature reviewed has primarily focused on the optimization of process performance through operating parameters. This includes temperature, mixing speed, oxidant dosage, and reaction time, which left aspects related to industrial implementation remaining underexplored. The studies have not reported energy consumption, equipment requirements, oxidant and catalyst costs, and process economics. Pilot-scale studies that assess the long-term operation and scalability of MAOD systems also remain limited. This highlights research gaps in the investigation of the techno-economic feasibility of MAOD to determine its suitability for commercial fuel desulfurization applications.

#### 3.4.5. Limitations of MAOD

Several limitations remain for MAOD, despite its promising sulfur conversion efficiencies. The process often relies on oxidants such as  $\text{H}_2\text{O}_2$  and catalytic systems that may require catalyst recovery and reuse to maintain economic and environmental sustainability. Catalyst regeneration, recovery, or replacement remains necessary as catalytic MAOD systems may experience catalyst deactivation, loss of active sites, and reduced activity over repeated cycles. These factors increase process complexity and affect the economic and environmental sustainability of the systems. The use of model fuels in the majority of the studies may not accurately represent the complexity of real fuel matrices, which means that the MAOD performance observed under these conditions may not be directly transferable to practical fuel systems.

The effectiveness of MAOD is also influenced by the balance between mass transfer and reaction kinetics. While high-intensity mixing can enhance interfacial contact between phases, improvements in mixing may not significantly increase sulfur conversion when the process becomes reaction-rate limited [50]. These factors should be considered when evaluating the practical implementation of MAOD systems.

## 4. Conclusions

The key research findings from the review can be summarized into three points. MAOD demonstrates high conversion efficiency achieving up to 100% sulfur conversion for both model fuel [31] and real fuel [33]. Operating parameters were also found to significantly affect the MAOD system performance, while MAOD effectively targets DBT and BT compounds that are resistant to conventional hydrosulfurization techniques.

MAOD systems commonly use  $\text{POM}/\text{H}_2\text{O}_2$  as the oxidant/catalyst which achieves high efficiency values from 97 to 100%. These values come from systems using DBT/BT-spiked toluene model fuel. Different oxidant/catalyst pairings have been explored as well as oxidant-only systems. The systems that employed oxidant-only systems exhibited a decrease in sulfur conversion. The crude Fe (VI) system used by Haboc et al. [23] exhibited only an 88% conversion for BT and the activated carbon supported HPW system used by

Barilla et al. [28] exhibited only a 62% conversion. Systems using real fuel were also tested where MAOD proved to be an efficient method for raw diesel fuel. A sulfur conversion value of 100% for real and model fuel is achievable using an HPW/H<sub>2</sub>O<sub>2</sub> system based on Lu et al. [33]. Using a different POM, Choi et al. [36] achieves a value of 96.6% conversion using an NaPW/H<sub>2</sub>O<sub>2</sub> system. A singular study proves that MAOD is a viable option for treating sour gas condensates by achieving a sulfur conversion of 96%. Pyrolysis oil was also tested using the crude Fe(VI) oxidant-only system and exhibited the lowest sulfur conversion studies in all the studies at 53–55% conversion.

In the parametric studies included in the review, it is observed that operating parameters greatly influence the performance of the MAOD system. The studies in the review had a temperature range of 40–70 °C, with higher temperatures exhibiting better sulfur conversions due to the faster reaction kinetics. Greater mass transfer between phases is also seen at systems operating at higher mixing speeds, and studies have adopted a baseline of 10,000 rpm. However, the parameter is limited by reaction kinetics. It is discovered that mixing speed can be rendered insignificant if the system has sufficient mass transfer already but was limited by slow reactions. Parametric studies had reaction times ranging from 3 min to 90 min and the common optimal reaction time is found at 30 min. Reaction time remained a significant parameter even in interaction with other variables as longer residence times resulted in higher conversion values. The effect of reaction time in efficiency of the system is however limited by the MAOD systems as studies that employed longer reaction time exhibited lower conversion values when using a different oxidant/catalyst system [28].

Overall, this paper reviewed a collection of studies regarding MAOD using a research matrix to provide a synthesis of what has been explored, trends in the research, and gaps to be addressed. The paper offers a perspective on MAOD as a potential method to treat fuel as compliance with various national standards and sustainability requirements. Studies that explicitly declare a maximum sulfur conversion and optimal operating parameters were included. The results of the review showed that MAOD achieves high conversion values for model and real fuel and proves it is a viable option. Operating parameters are significant in achieving efficient system performance. Optimization studies using RSM showed that baseline operating conditions of 10,000 rpm, 70 °C, and 30 min reaction time can achieve 85–100% sulfur conversion. The effects remain limited by the oxidant/catalyst system and the reaction kinetics of the conversion. Gaps regarding the complexity of real fuel in their components and the lacking comparative studies to show how close model fuels can simulate real fuel are found. Design methodologies are also limited to RSM, which suggests that exploration of other methodologies is needed. Overall, MAOD as a methodology to treat fuel is a promising technology that shows potential in achieving high conversion values, but research is limited to laboratory scale experiments using simple mixtures used as model fuel. The study offers a strong perspective to help future research direction. Comparative studies and parametric studies using more complex fuel mixtures are recommended.

**Author Contributions:** Conceptualization, M.P.M., C.A.C. and A.E.S.C.; Formal analysis, E.J.T. and L.J.D.; Supervision, M.P.M., C.A.C. and A.E.S.C.; Writing—review and editing, E.J.T., L.J.D., M.P.M., C.A.C. and A.E.S.C. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by the Department of Science and Technology (DOST) of the Philippines through its Engineering Research and Development for Technology (ERDT) program, the National Research Council of the Philippines Grants-in-Aid (NRCP-GIA FY 2025 Project No. G-85), and the Research and Grants Management Office (RGMO), De La Salle University (Project No. 10 R R 1TAY25-3TAY25). The APC was funded by De La Salle University Science Foundation, Inc.

**Data Availability Statement:** No new data were created or analyzed in this study. Data sharing is not applicable to this article.

**Conflicts of Interest:** The authors declare no conflicts of interest.

## Abbreviations

The following abbreviations are used in this manuscript:

|      |                                           |
|------|-------------------------------------------|
| MAOD | Mixing-assisted Oxidative Desulfurization |
| BT   | Benzothiophene                            |
| DBT  | Dibenzothiophene                          |

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