

A Review on the Current and Potential Oxidant-Catalyst Systems in Mixing-Assisted Oxidative Desulfurization

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The release of sulfur-containing compounds into the atmosphere has been a widespread environmental issue throughout the world. In producing fuel oils, oxidative desulfurization (ODS) has been a widely studied alternative method to hydrodesulfurization, as it requires lower operating parameters. Mixing-assisted oxidative desulfurization (MAOD) is a modification of the ODS process that employs high-shear mixing to enhance fluid/fluid interfacial area between an oxidant and a fuel. Since MAOD is a relatively new field, not all common ODS oxidants and catalysts have been applied to the process. This paper reviews common oxidant-catalysts used in the MAOD process, namely, H₂O₂ and Fe(VI) oxidants, along with heteropoly acids (HPA) and acetic acid catalysts. The majority of MAOD studies have shown that H₂O₂ is commonly preferred as it is cheap, easily available, and environmentally friendly. HPA consists of polyoxometalate anions that create intermediate products in reaction with H₂O₂ to enhance oxidation. A highly effective HPA in MAOD studies is phosphotungstic acid (HPW). Optimization studies that used the H₂O₂-HPW oxidant/catalyst system reported sulfur conversions ranging from 82 % and above. The highest sulfur removal of 100 % was achieved at 40 °C operating temperature, 10,000 rpm agitation speed, and 1:1 PTA to catalyst ratio for this system. In addition, the incorporation of activated carbon to HPW was reported to enhance the desulfurization performance, with the AC acting as a catalyst and an adsorbent. On the other hand, Fe(VI) has a high redox potential among commonly utilized oxidants in ODS. Fe(VI) is usually paired with an acetic acid catalyst as the high redox potential is achieved at acidic conditions. However, the separation and recycling of acetic acid after desulfurization has been seen as a potential challenge. With this, ODS studies have paired Fe(VI) with solid superacid, such as SO₄²⁻/γ-Al₂O₃, for their highly acidic strength, and environmental friendliness. An optimization study reported sulfur removal of 91.3 % using a Fe(VI)-SO₄²⁻/γ-Al₂O₃ system and 100 % using H₂O₂/UIO-66(Zr) system. These systems can be subjected to MAOD for a potential increase in sulfur conversion. Applying further optimization to lower economic and environmental implications may be advantageous for possible scale-up applications of the MAOD process.

1. Introduction

The atmospheric emission of sulfur compounds has been a rampant environmental problem around the world. When combined with moisture and other pollutants, sulfur dioxide causes corrosion and strengthens acid rain (US EPA, 2019). A primary source of non-natural sulfur dioxide is fossil fuel combustion. In effect, regulations have been set to limit the concentration of sulfur compounds in fuels. In the Philippines, the maximum sulfur content in fuel is limited to 50 ppm (DENR, 2015). Meanwhile, neighboring countries such as Taiwan and Japan have set their standards to 10 ppm to 15 ppm for both diesel and gasoline (Taiwan EPA, 2020).

In the petroleum refining industry, the most common operation to reduce sulfur concentration in transportation fuel is hydrodesulfurization (HDS). HDS utilizes hydrogen to break the bond between the sulfur atom and the hydrocarbon to produce hydrogen sulfur (Haghighi and Gooneh-Farahani, 2020). However, the operating requirements of HDS bring disadvantages to the process cost, as it typically requires 300-400 °C, 3-6 MPa, and a high catalyst volume to operate (Sikarwar et al., 2019). HDS is also ineffective in removing refractory organosulfur compounds, which constitute a major percentage of the total sulfur content in fuel oils (Rajendran et al., 2020).

Due to the economical requirements of HDS, other methods of desulfurization have been widely studied by researchers. One of these novel methods is oxidative desulfurization (ODS). ODS has two main advantages over HDS. ODS can be performed at mild temperature and pressure to operate (Choi et al., 2021). It can also oxidize refractory organosulfur compounds which are difficult to remove through HDS, such as benzothiophene (BT) and dibenzothiophene (DBT) (Wan et al., 2014). The reactions in this method are heterogeneous in general, with two or more immiscible phases involved. The mechanism of ODS works by oxidizing the refractory sulfur-containing compounds into their corresponding sulfoxides and sulfones. After this, the sulfones are removed by either extraction or adsorption due to their increased polarity (Rajendran et al., 2020). Catalysts are greatly used in ODS as studies have shown that they selectively promote sulfur oxidation and consequently increase reaction rate. However, the design of the catalyst is also crucial for selective oxidation to work. A significant part of this designing is the recyclability of catalysts to further promote green technology application in fuel desulfurization (Chen and Yuan, 2022).

A modification on the ODS process called the mixing-assisted oxidative desulfurization (MAOD) employs high-shear mixing to increase the rate of sulfur conversion. As MAOD is a relatively recent area of study, not all commonly used ODS oxidant and catalyst systems have been tested for their oxidation effectiveness in combination with the process. To this date, there are no known review papers summarizing MAOD optimization studies and their oxidant/catalyst systems. With this, the paper aims to report the common oxidants used in the MAOD process, namely, H_2O_2 and ferrate, along with heteropoly acids (HPA) and acetic acid catalysts. Potential MAOD catalysts which are tested in ODS are discussed as well.

2. Mixing-assisted oxidative desulfurization

MAOD utilizes high-shear mixing to create an emulsion of the fuel and oxidant. This speed enhances fluid/fluid interfacial area between the oxidant and fuel and consequently enhances mass transfer (Choi et al., 2016b). Studies have shown that increasing the mixing speed can significantly improve sulfur conversion. One of the early kinetic studies in MAOD reported that increasing mixing speed from 5,500 rpm to 10,000 rpm increased the reaction rate from 0.0891 min^{-1} to 0.1528 min^{-1} in a pseudo-first order kinetic model. This translates to an increase of sulfur conversion from 92 % to 99 % (de Luna et al., 2017).

Phase transfer agents (PTA) are also utilized since the sulfur compounds are in the organic phase and the oxidants are in the aqueous phase. PTAs are able to incorporate with the oxidants and transfer them to the organic phase by decreasing their polarity. Aside from PTAs, catalysts are also incorporated to decrease the reaction time of the process (de Luna et al., 2017).

A summary of SCOPUS-indexed MAOD optimization studies is presented in Table 1, together with the corresponding target sulfur compounds, oxidants, catalysts, operating parameters and maximum sulfur conversion. It is worthy to note that the majority of these studies used hydrogen peroxide (H_2O_2) as an oxidant. H_2O_2 is one of the preferred oxidants in ODS as it is cheap, easily available, and environmentally friendly as it produces only water as a by-product (Sikarwar et al., 2019). Meanwhile, ferrate (Fe(VI)) has the highest redox potential in acidic medium, among the commonly utilized oxidants in ODS (Jiang, 2007).

2.1 Current oxidant-catalyst systems

H_2O_2 has a 47 % amount of active oxygen per mass unit, is highly selective and produces a good product quality. However, this compound can cause the ignition of flammable materials at high concentrations (Mirshafiee et al., 2023). The earliest MAOD studies which utilized H_2O_2 were the study of Lu et al. (2014) and Wan et al. (2014). Lu et al. (2014) compared the application of ultrasonic radiation and high-shear mixing in ODS. Prior to this, ODS primarily utilized magnetic stirring which resulted to low sulfur conversion. The results of the study showed that both systems were able to reach 99 % DBT and 98 % BT conversion. Optimized reaction conditions for MAOD were reported to be 10,000 rpm mixing speed, 70 °C reaction temperature, and 30 min reaction time. Meanwhile, the study of Wan et al. (2014) compared two MAOD arrangements: continuous high-shear mixing and in-line mixing. The study revealed that in-line high-shear mixing is the more economical choice between the two arrangements due to the handling of a larger volume of fuel. With the same sulfur removal of 82 %, the in-line mixer was able to handle a flow rate of 500 mL/min compared to the 360 mL/min capacity of the continuous high-shear mixing.

de Luna et al. (2018a) explored the integration of adsorptive desulfurization in MAOD by utilizing raw diesel fuel with an initial sulfur content of 1,480 ppm and comparing CSTR and in-line high-shear mixing arrangements. Results revealed that the utilization of the in-line mixer produced better oxidation results. Similar to the results of Wan et al. (2014), this type of mixer was also more favorable as it was able to treat a larger capacity of diesel than CSTR. Meanwhile, de Luna et al. (2018b) utilized a commercial diesel with initial sulfur concentration of 1,428 ppm. The study tested the effectivity of chitosan-coated bentonite and activated carbon as adsorptive

materials for sulfones produced from MAOD. Results revealed that the chitosan-coated bentonite had a more powerful absorption capacity of 4.72 mg/g than that of activated carbon, which was 3.73 mg/g.

From the studies that utilized H_2O_2 as an oxidant, the study of de Luna et al. (2017) and Wan et al. (2019) reported the highest sulfur conversion of 100 %. This was achieved using the following operating parameters: 400 °C calcination temperature, 30 min reaction time, and 1:2 PTA:HPW molar ratio (Wan et al., 2019); 40 °C temperature, 10,000 rpm mixing speed, and 1:1 PTA:catalyst ratio (de Luna et al., 2017). The latter study reported a better catalytic performance using a lower temperature and without using calcination. This indicates that calcination of HPW is not significant in enhancing its catalytic performance in ODS of DBT (Wan et al., 2019).

On the other hand, the rest of the papers that utilized H_2O_2 oxidant had a maximum sulfur removal range from 62.73 % to 99 %. Furthermore, these systems were operated at atmospheric pressure and 40 °C to 80 °C temperature range. This proves that the MAOD process is effective in converting sulfur compounds at ambient pressure and significantly lower temperatures than the requirement of the HDS process. Furthermore, Barilla et al. (2022) reported that increasing the temperature is favorable to the oxidation activity. This is due to the rapid movement of molecules which promotes easier mixing.

Heteropoly acids are also common catalysts used along with H_2O_2 . They are complex acids composed of polyoxometalate anions, made up of metal-oxygen octahedra, and protons (Wang et al., 2010). HPAs have stable structure, good thermal stability, and are considered as a green catalyst (Li et al., 2020). In the listed studies in Table 1, the HPA that is commonly used is phosphotungstic acid (HPW) with a chemical formula of $\text{H}_3\text{PW}_{12}\text{O}_{40}$.

The studies of Choi et al. (2016b) and Choi et al. (2016c) have compared various HPAs in the frame of MAOD. Both studies have shown that HPW was used to achieve highest sulfur conversions up to 97.4 %. Metal derivatives react with H_2O_2 to form peroxocomplexes, which are stronger in oxidation and highly selective. The quaternary ammonium cation of the PTA binds with the peroxocomplexes and transfers to the organic phase. The selective oxidation of the sulfur compounds then occurs, forming sulfones. Following this, the peroxocomplexes are reduced and return to the aqueous phase with the dissociation with PTA (Choi et al., 2016b). In addition, supported HPAs used in ODS are being applied to MAOD. Activated carbon has been reported to improve the stability and effectiveness of HPAs (Barilla et al., 2022).

However, no studies have explored the recyclability of HPAs in MAOD. The small specific surface area of HPAs limit their recyclability, however, this can be improved by dispersing the catalyst on support with high specific area (Li et al., 2020).

Another oxidant which is used in an MOAD study is Fe(VI). Fe(VI) is commonly utilized in ODS due to its high reduction potential of 2.2 V in acidic media and environmentally friendly reaction products (Jiang, 2007). Acetic acid is commonly used as a catalyst with Fe(VI) because of this characteristic. A protonated ferrate form, $[\text{Fe}(\text{O})_3(\text{OH})]^-$, produced in the reaction was observed to be a more effective oxidant than Fe(VI) alone, with an increasing oxidation power as pH is lowered (Xie et al., 2021).

Based on Table 1, the study of Choi et al. (2016a) is the sole paper that explored the effectivity of Fe(VI) as an oxidant in the MAOD of sulfur compounds and real diesel oil. The proponents considered the following operating parameters in the optimization of the process: agitation speed (7,600 rpm to 14,000 rpm), temperature (50 °C to 70 °C), and mixing time (10 min to 30 min). Optimal parameters were reported to be: 12,198 rpm, 52.22 °C, and 15.42 min for BT and 8,704 rpm, 51.26 °C, and 14.43 min for DBT. Maximum conversion for BT and DBT reached 84.35 % and 93.68 %. The paper also reported a significant finding in the effect of reaction temperature in the system, wherein a high temperature is favorable in forming Fe(VI) complexes. However, a further increase in temperature to 70 °C may lower oxidation activity due to the low thermostability of Fe(VI) at this temperature. Despite the strong oxidation capacity of Fe(VI), one of the difficulties in using the oxidant is its stability at lower pH. In this condition, Fe(VI) may undergo self-decomposition and react with water to form Fe(III), oxygen gas, and hydroxide ion (Xie et al., 2021).

From the studies listed in Table 1, the study of Pouladi et al. (2019) solely used a gas-liquid system in MAOD without a catalyst. The reaction conditions were ambient pressure, ambient temperature, 5000 rpm mixing speed, and 30 min reaction time. The study used a combination of H_2SO_4 , HNO_3 , and NO_2 as oxidizing agents and optimized total sulfur reduction by varying oxidant concentration. The combination of H_2SO_4 and HNO_3 produces a nitronium cation (NO_2^+), which is a stronger oxidizing agent than the two compounds alone. A 1500 g sour gas condensate sample with 2300 ppm total sulfur content. Optimization via response surface methodology resulted to a final total sulfur concentration of 102 ppm or 95.56 % sulfur removal using 0.682 mol HNO_3 , 0.264 mol NO_2 , and 0.593 mol H_2SO_4 . It was also reported that HNO_3 concentration had the most significant effect in sulfur removal compared to the other oxidizing agents. Meanwhile, a mechanism between H_2SO_4 and NO_2 was reported, where H_2SO_4 acts like a catalyst and NO_2 acts as the stoichiometric reactant.

Table 1: Scopus-indexed MAOD optimization studies from 2014

Authors (Year)	Target Sulfur Compounds	Oxidant	Catalyst	Maximum Sulfur Conversion and Operating Parameters
Barilla et al. (2022)	BT, DBT	H ₂ O ₂	Activated carbon supported phosphotungstic acid	62.73 % sulfur removal Mixing speed: 16,800 rpm Mixing time: 88.5 min Temperature: 63.28 °C
Wan et al. (2019)	DBT	H ₂ O ₂	Calcinated phosphotungstic acid	100 % sulfur conversion Calcination temperature: 400 °C Reaction time: 30 min PTA:HPW molar ratio: 1:2
Pouladi et al. (2019)	Sulfur compounds in sour natural gas condensate	H ₂ SO ₄ , HNO ₃ , and NO ₂	None	95.56 % conversion 0.682 mol HNO ₃ 0.264 mol NO ₂ 0.593 mol H ₂ SO ₄
de Luna et al. (2018b)	Sulfur compounds in diesel fuel	H ₂ O ₂	phosphotungstic acid	81.97 % conversion Mixing speed: 12,000 rpm Temperature: 80 °C Mixing time: 34 min
de Luna et al. (2018a)	BT, DBT in raw diesel fuel	H ₂ O ₂	phosphotungstic acid	85.90 % sulfur removal Temperature: 70 °C Agitation speed: 18,000 rpm Diesel flow rate: 500 mL/min Oxidant flow rate: 300 mL/min
de Luna et al. (2017)	DBT	H ₂ O ₂	phosphotungstic acid	100 % conversion Temperature: 40 °C Agitation speed: 10,000 rpm PTA:catalyst ratio: 1:1 ratio
Choi et al. (2016c)	BT, DBT	H ₂ O ₂	sodium phosphotungstate, phosphotungstic acid, phosphomolybdic acid, and silicotungstic acid	94.8 % DBT and 97.4 % BT sulfur conversion Temperature: 70 °C Mixing time: 30 min Using sodium phosphotungstate
Choi et al. (2016b)	BT, DBT	H ₂ O ₂	phosphotungstic acid, phosphomolybdic acid, and silicotungstic acid	>90 % using phosphotungstic acid
Choi et al. (2016a)	BT, DBT	Fe(VI)	Acetic acid	84.35 % BT conversion Temperature: 52.22 °C Agitation speed: 12,198 rpm Mixing time: 15.42 min 93.68 % DBT conversion Temperature: 51.26 °C Agitation speed: 8,704 rpm Mixing time: 14.43 min
Lu et al. (2014)	BT, DBT	H ₂ O ₂	phosphotungstic acid	99 % DBT and 98 % BT conversion Mixing speed: 10,000 rpm Temperature: 70 °C Mixing time: 30 min
Wan et al. (2014)	Sulfur compounds in diesel fuel	H ₂ O ₂	phosphotungstic acid	98 % removal Mixing speed: 18,000 rpm Diesel flow rate: 500 mL/min H ₂ O ₂ flowrate: 300 mL/min

2.2 Potential oxidant-catalyst systems

As MAOD is a relatively new field of study, there are many potential oxidant-catalyst systems that can be applied to it. In recent years, zirconium metal-organic frameworks (Zr-MOFs) have gained interest due to their high surface area, porosity, and strong interactions with oxygen (Ramsahye et al., 2014). In the paper of Zhang et al. (2017), the UIO-66(Zr) catalyst was synthesized and applied to the ODS of a model oil containing sulfur compounds. The experiment was performed using an H_2O_2 oxidant at 30 to 70 °C under constant stirring and reflux. Results showed that sulfur conversion reached up to 100 %, proving that the system was effective for ODS. However, the recyclability of the catalyst was found to be inefficient, as the conversion decreased to 50 % after five cycles. In the study of Wang et al. (2021), phosphomolybdic acid was niched in UIO-66 to improve its performance. Results showed that the synthesized catalyst was able to remove all DBT in the model fuel after 60 min. The stability of the catalyst was also studied through recycling. After 10 cycles, the system was able to remove 92.10 % of DBT, proving that good catalyst stability.

Another possible system covers the use of sulfated metal oxide catalysts with Fe(VI). Naghavi et al. (2021) studied the ODS of sulfur compounds using Fe(VI) derived by electrolyzing scrap iron in conjunction with $SO_4^{2-}/\gamma-Al_2O_3$. Successful sulfate impregnation was determined through the detected increase of sulfur concentration via X-ray fluorescence spectrometer. Based on the study, DBT removal reached up to 91.3 % using the ferrate solution, catalyst, and an extraction solvent. The suggested reaction pathway indicated that Fe(VI) is protonated at the acidic sites of the catalyst. Then, DBT is adsorbed over the catalyst before it is oxidized by the protonated Fe(VI). The last step is the extraction of the sulfone via the solvent. The study also evaluated the recyclability of the catalyst after recovery. It was reported that DBT removal reached up to 80.1 % after 4 recycling stages. The decrease was attributed to the accumulation of water and sulfone products in the catalyst. However, this is still beneficial compared to other Fe(VI) catalysts, such as acetic acid, which is hard to recover after usage.

3. Conclusion

MAOD is a novel modification of the ODS process involving high-shear mixing to enhance the mass transfer between an oxidant and the target sulfur compounds in the fuel. This work highlights MAOD optimization studies and various oxidant/catalyst systems that were used in these works. Based on the literature review, the majority of MAOD studies utilize H_2O_2 and phosphotungstic acid as an oxidant/catalyst system. In addition, the use of Fe(VI) together with an acetic acid catalyst had also been applied in MAOD. From these studies, a maximum of 100 % sulfur conversion was achieved in ambient conditions. Additionally, promising catalysts applied in ODS, such as Zr-MOFs and $SO_4^{2-}/\gamma-Al_2O_3$, have exhibited high sulfur conversion and recyclability properties. These oxidant-catalyst systems can potentially be applied to MAOD as basis for future research. These systems may also improve the development of cost-effective and environmentally friendly desulfurization techniques.

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