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Advanced Desulfurization via Ultrasonic-Assisted Oxidative Methods in Gasoline and Crude Oil Towards Sustainability

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The presence of sulfur in crude oil and gasoline is crucial in both environmental and industrial contexts. This paper explores the intricate nature of sulfur, including elemental sulfur, hydrogen sulfide, carbonyl sulfide, and various organic compounds like thiophene derivatives. Effective sulfur management is essential due to its harmful effects on oil processing catalysts and the environment, leading to air pollution and acid rain. Reviewing environmental regulations mandating a minimal sulfur limit of 10 ppm in gasoline emphasizes the urgent need for enhanced desulfurization methods to address the adverse impacts of burning high-sulfur fuels on the environment and fuel efficiency. The review delves into Ultrasonic-Assisted Oxidative Desulfurization (UAOD), an innovative technique that maximizes sulfur removal from petroleum products. This process harnesses the power of ultrasonic waves to elevate heterogeneous reactions and optimize the effectiveness of oxidants, increasing the overall efficiency of sulfur removal. UAOD is a promising method for addressing sulfur-related challenges within the petroleum sector, as it yields enhanced desulfurization results and operational efficacy. Response Surface Methodology (RSM) efficiently optimizes parameters across various manufacturing procedures, including desulfurization studies, using designs like Box-Behnken or central composite. RSM effectively optimizes reactions affected by independent variables, demonstrated in the desulfurization of diesel and gasoline using methods like Mixing-assisted Oxidative Desulfurization (MAOD) and UAOD with NaPW/H_2O_2.

1. Introduction

Fossil fuels have had a substantial influence on humanity and the environment. The consumption of fossil fuels has long been a cornerstone of global energy production, powering industries, transportation, and daily life. However, the widespread use of fossil fuels comes with significant environmental challenges, particularly sulfur emissions. Sulfur compounds present in crude oil and gasoline contribute to air pollution, acid rain formation, and adverse health effects, prompting regulatory agencies worldwide to impose stringent standards for sulfur content in fuel products. To address the issue of sulfur emissions, Clavin (2017) notes that oil refineries use extensive procedures to remove the majority of sulfur from fuel, bringing it down to the level that is mandated by the government. The process of diminishing the sulfur content in fuels is called desulfurization. This is regarded as an important step in fuel utilization because it allows for advanced emission controls and reduces the environmental impact of sulfur. Desulfurization has become a popular method of obtaining ultra-clean fuels. One innovative method in the industry is Ultrasonic-Assisted Oxidative Desulfurization (UAOD). UAOD is a technique that uses ultrasonic waves that can increase heterogeneous reactions, boost the activity of oxidants, and hasten the breakdown of macromolecular molecules. It can enhance desulfurization efficiency through the promotion of molecular interaction and the generation of free radicals (Lin et al., 2020). The optimization of desulfurization processes is essential for maximizing efficiency and minimizing operational costs. The advantage of UAOD in terms of cost-effectiveness is that it is a better option for refining economics due to the lower operating temperature and pressure it requires to desulfurize, reducing energy consumption costs (Chen et al., 2013). Response Surface Methodology (RSM) (Chelladurai et al., 2020) has emerged as a valuable tool for optimizing parameters in various manufacturing processes, including desulfurization studies. Techniques

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such as the Box-Behnken design (Rao and Kumar, 2012) and central composite design (Wagner et al., 2013) enable researchers to systematically explore the effects of independent variables on desulfurization efficiency and identify optimal operating conditions.

This review uniquely focuses on UAOD and RSM for desulfurization optimization, providing novel insights into these advanced techniques. Unlike conventional reviews, it specifically delves into their potential for enhancing sulfur removal efficiency and operational effectiveness. Through a thorough analysis of recent literature, it aims to advance our understanding of modern desulfurization strategies and their industrial implications. The approach utilized to gather pertinent literature for this review article involved a thorough exploration of academic repositories such as ScienceDirect, ResearchGate, and Google Scholar. Specific keywords such as "ultrasonic assisted oxidative desulfurization," "methods for desulfurization," "response surface methodology," and "analysis of sulfur composition" were employed to locate relevant research. Citation tracking was utilized to investigate citations within identified articles for additional pertinent resources. A wide range of studies covering key aspects of the subject matter were compiled to offer a comprehensive comprehension and evaluation in the review paper.

2. Sulfur Composition Analysis

Crude oil, a liquid petroleum product, contains hydrocarbons like paraffin, alkylbenzenes, and aromatics, along with heteroatomic compounds such as sulfur, carbon dioxide, oxygen, and nitrogen. Fahim et al. (2010) noted sulfur concentrations in crude oil ranging from approximately 0.05 wt% to over 10 wt%, typically falling between 1-4 wt%. Sulfur exists in various forms in crude oil, including elemental sulfur (S), dissolved hydrogen sulfide (H_2S), and carbonyl sulfide (COS), with organic forms being the most significant. Gasoline, derived from crude oil and various petroleum products, varies in properties depending on composition, refining processes, and geographical origin of the crude source. Zhang et al. (2010) analyzed gasoline sulfur content in China, finding that most samples (88 %) were below 500 ppm, with 41 % meeting or falling below 150 ppm, aligning with China III gasoline standards. USEPA (2016) regulations, specifically Tier 3, aim to enhance air quality by limiting sulfur content in gasoline. The annual average sulfur limit is set at 10 ppm, with a maximum per-gallon limit of 80 ppm. Oxygenates, like denatured fuel ethanol, blended into gasoline, should contain no more than 10 ppm sulfur per gallon and consist solely of carbon, hydrogen, nitrogen, oxygen, and sulfur. Denatured fuel ethanol should also not exceed 3 % denaturants, which must be certified gasoline, gasoline blend stocks, or natural gas liquids. Parties subject to Tier 3 regulations include gasoline refiners and importers, oxygenate producers and blenders, certified ethanol denaturant producers and importers, and gasoline additive manufacturers. Achieving extremely low levels of sulfur in gasoline presents technical obstacles such as the control of catalytic fines and the maintenance of ideal viscosity, all while meeting increasing market requirements in the face of diminishing feedstock quality (Kjellström, 2021). UAOD addresses these challenges by selectively eliminating sulfur compounds without generating fines, achieving thorough desulfurization, and enhancing stability and lubricity, providing an efficient alternative to meet demand (Mei et al., 2003).

Desulfurization of petroleum fractions is crucial to prevent damage to processing catalysts and minimize environmental impacts (Calin et al., 2021). Various sulfur compounds, including benzothiophenes (BT) and dibenzothiophenes (DBT), are commonly found in crude oil and must be eliminated through techniques such as hydrodesulfurization (HDS) (Schou and Myhr, 1988). BT is considered the easiest to oxidize and is followed by DBT and then 4,6-dimethyldibenzothiophene (4,6-DMDBT) (Shafi & Hutcfhings, 2000). This difference is primarily due to the presence of bulky aromatic groups near the sulfur atom in DBT and 4,6-DMDBT. These groups create steric hindrance, making it physically more difficult for the oxidizing agent to access and react with the sulfur atom. These derivatives interact with oil processing catalysts primarily through mechanisms such as reactive adsorption, selective adsorption, and π-complexation. Understanding these interactions informs desulfurization strategies by guiding the selection of catalysts with specific metal ions and properties tailored to enhance sulfur compound adsorption and selectivity (Dehghan and Anbia, 2017). The effectiveness of desulfurization processes is critical for complying with environmental standards and maintaining the quality of refined petroleum products. The use of optimization techniques like RSM is gaining prominence in desulfurization research (Zhou et al., 2020). RSM allows for the efficient optimization of operating variables to enhance desulfurization efficiency while minimizing costs (Eyjolfsson, 2015). By leveraging RSM, researchers can develop empirical models and identify optimal conditions for desulfurization processes, contributing to advancements in sulfur management strategies within the petroleum industry. In summary, the regulation of sulfur content in petroleum products, coupled with the development of effective desulfurization methods and optimization techniques like RSM, underscores the importance of addressing sulfur-related challenges for environmental sustainability and industrial operations.

3. Ultrasonic Assisted Oxidative Desulfurization

UAOD efficiently lowers sulfur in marine fuels to meet low sulfur regulations by optimizing ultrasonication and mechanical stirring for higher conversion rates (Houda et al., 2021). Additionally, UAOD excelled in reducing sulfur in Iranian heavy crude oil, especially with $CuSO_4$ as the PTA, and showed significant improvement in multistage extraction for enhanced sulfur removal (Ghahremani et al., 2021). This section provides a detailed examination of the historical context of UAOD and explores the various factors that impact its effectiveness. It also outlines the adverse environmental consequences associated with this process, its underlying mechanism, factors that influence its efficacy, and the experimental approach used in its study.

3.1 Negative Environmental Effects of Contaminated Fuels

Acid rain, building corrosion, harm to forests and agriculture, poisoning of catalytic converters, acidification of lubricating oil, and decreased fuel efficiency are a few of the negative consequences of using contaminated fuels. Compounds containing sulfur aggravate these problems by decreasing the API gravity and further lowering the grade of petroleum products. H_2S is the main difficulty during crude oil extraction, storage, transportation, and processing. H_2S poses a significant challenge throughout the entire process of crude oil extraction, storage, transportation, and processing, stemming from activities related to oil and gas exploration, extraction, processing, and post-processing. This compound presents a tangible risk to workers, particularly when attention lapses occur, potentially leading to poisoning incidents. It is imperative to mitigate sulfur-containing chemicals to safeguard the well-being of humans, protect the environment, and ensure the integrity of production equipment (Ja'fari et al., 2018).

3.2 Mechanism of UAOD

Various desulfurization techniques have been developed to comply with the established environmental and safety regulations governing the utilization of gasoline and crude oil, among which Ultrasonic-Assisted Oxidative Desulfurization (UAOD) stands out. Numerous challenges are associated with achieving full mixing of water and oil phases using conventional mechanical agitation in oxidative desulfurization processes (Lin et al., 2020). The application of ultrasonic waves demonstrates remarkable efficacy in enhancing molecular interaction and facilitating the formation of microemulsions between the two phases. This phenomenon generates radicals, induces local high pressure and temperature conditions, and synergizes with the heat and mechanical forces of the ultrasonic wave. UAOD effectively oxidizes sulfide compounds, optimizes the oxidizing capacity, enhances reaction selectivity, and significantly reduces the reaction time. The method alters the process dynamics and orientation, enabling complete oxidation reactions to occur. During the extraction phase, ultrasonic intervention ensures thorough mixing between the extractant and the gasoline or crude oil, promoting optimal interaction between the extractant and the oxidized sulfide constituents.

3.3 Factors that Influence UAOD Efficacy

The efficacy of Ultrasonic-Assisted Oxidative Desulfurization (UAOD) is subject to various influencing factors that can lead to deviations in outcomes. The performance of UAOD is dependent on the frequency of ultrasonic waves employed (Ja'fari et al., 2018). In UAOD, ultrasonic waves create cavitation and emulsification, improving contact between oil and oxidant for faster sulfur removal (Khodaei et al., 2018). The desulfurization efficiency depends on the frequency, power, irradiation time, temperature, oxidant, and the type of oil being treated (Zhou et al., 2020). Optimizing these factors is key to maximizing the effectiveness of UAOD. Lower frequencies generate fewer cavitation bubbles of larger resonant sizes compared to higher frequencies. Optimal organic matter removal in the aqueous phase is observed within the range of 200–850 kHz. Extending the reaction time can enhance efficiency by facilitating improved contact between immiscible phases. This allows sulfur-containing polar oxidants to permeate the aqueous phase. The UAOD process accelerates the production of sulfoxides and sulfones by generating a higher concentration of excited radicals. The choice and concentration of the oxidizing agent influence the conversion of sulfur-containing compounds into sulfoxides and sulfones. Ensuring the optimal concentration of oxidant facilitates efficient desulfurization while mitigating excessive oxidant consumption or byproduct formation. The composition of crude oil and gasoline exerts a significant influence on UAOD performance. Crude oils characterized by high sulfur content may necessitate more stringent operating conditions for effective desulfurization. Additionally, the presence of contaminants such as nitrogen and metals can alter the kinetics and efficiency of UAOD reactions (Houda et al., 2018).

3.4 Experimental Methodology for UAOD

A DS-3510DTH type ultrasonic reactor operating at a frequency of 40 kHz was utilized in conjunction with a TSN2000 type fluorescence sulfur nitrogen analyzer for the comprehensive analysis of crude oil samples, oxidants, and demulsifiers in the investigation of the UAOD of crude oil (Lin et al., 2020). The experimental setup initially involved mixing raw materials in a conical flask to replicate the sample conditions. The response proceeded after a predetermined time and the reaction liquid was cleaned and separated after the reaction. The reaction liquid was then subjected to thorough cleaning and separation procedures. A digital constant temperature magnetic stirrer facilitated the oxidation desulfurization experiment while a fluorescence sulfur nitrogen meter gauged the sulfur content in the reaction oil. The use of UAOD yielded significantly superior reductions in sulfur content compared to Oxidative Desulfurization (ODS). The optimal conditions for UAOD efficacy were obtained at a temperature of 65 °C, a reaction duration of 10 min, an oxidant dosage of 200 ppm, and a demulsifier dosage of 60 ppm. This contributed to a relatively high desulfurization rate of 65.28 %. The study shows that the role of ultrasonic waves in augmenting heterogeneous reactions is important in enhancing oxidant efficiency and facilitating the degradation of macromolecular compounds. Recent advancements in UAOD research extend beyond the limitations of traditional experimental methods. Researchers are developing highly active, microscopic nano-catalysts for a surface area boost under ultrasound, improving efficiency (Vafaee et al., 2021). Additionally, in-situ catalyst generation techniques are emerging, simplifying the process by creating catalysts directly within the reaction mixture (Mei et al., 2003).

4. Response Surface Methodology

This section delves into RSM, offering a comprehensive overview and experimental designs and examining its diverse applications in engine optimization and clean fuel production.

4.1 Overview of RSM and Experimental Designs

A compelling method used in optimizing parameters in different manufacturing procedures, which centers on mathematical and statistical techniques, can be obtained using the RSM. As fewer experiments are required to study a given element or level, this technique is usually employed in experiment designs (Chelladurai et al., 2020). Some of the common types of RSM design of experiments in desulfurization studies employ the use of the full factorial design, Box-Behnken design, or central composite design. A hypercube in an n-dimensional design space can be modeled using the full factorial design by defining the maximum and minimum values of each experimental factor (Sahoo and Barman, 2012).
The number of experiments in a full factorial design is decided by the number of factors and levels in the experiment. The number of levels of each factor is raised to the number of factors to obtain the number of experiments. However, the full factorial design is often hard to implement due to the high number of replications needed to analyze the data. Higher-order response surfaces can be generated using Box-Behnken models (Rao and Kumar, 2012). The Box-Behnken model is not based on factorial designs and requires fewer runs when compared to a normal factorial technique. As an independent quadratic design, the use of the model allows the study of the effects of a certain factor in an experimental design sequentially as long as the other factors are maintained at a constant level (Ait-Amir et al., 2020). Another method that is commonly used for generating second-order polynomials for the response variables in RSM is the Central Composite Cesign (CCD). The CCD is based on a two-level factorial design (Wagner et al., 2013). When comparing the three designs, the Box-Behnken model is considered suitable for experiments with a low number of replications as the experimental data employing the model can be effectively analyzed with fewer runs when compared to the full factorial and central composite designs.

4.2 Applications of RSM in Desulfurization and Clean Fuel Production

Table 1 provides a summary of the review of various applications of RSM in the field of oxidative desulfurization. The study of Barilla et al. (2022) focuses on the application of RSM in the field of mixing-assisted oxidative desulfurization using hydrogen peroxide as an oxidant. Zhou et al. (2020) enhanced UAOD efficiency in gasoline and crude oil. Choi et al. (2022) optimized ultrasound-assisted oxidative desulfurization in untreated diesel oil. Ghahremani et al. (2021) studied process parameters in UAOD of Iranian heavy crude oil using hydrogen peroxide (H_2O_2). Barilla et al. (2022) investigated clean fuel production security by desulfurizing diesel using mixing-assisted oxidative desulfurization (MAOD) with an activated carbon catalyst, phosphotungstic acid. They employed face-centered cubic design (FCC) in RSM to optimize MAOD parameters. Analysis conducted before and after MAOD treatment revealed diesel properties were maintained, promising future applications for the process. optimized desulfurization of gasoline and crude oil using RSM. Their findings suggest ultrasound treatment is more effective for crude oil, highlighting its potential for oil desulfurization. These findings contribute valuable insights into the application of ultrasonic treatment for oil desulfurization. Choi et al. (2022) investigated optimizing UAOD with NaPW/H_2O_2 to enhance clean fuel production sustainability. Their study emphasized the significance of ultrasound time, amplitude, catalyst dosage, and temperature in sulfur conversion efficiency. Using RSM via Box-Behnken design, they determined essential variables impacting sulfur conversion. Achieving a theoretical value of 99.6 % and a verified value of 95.0 % sulfur conversion in diesel, they demonstrated the efficiency of UAOD as an industrial desulfurization technology. Ghahremani et al. (2021) highlight the efficiency of UAOD in reducing sulfur levels in Iranian heavy crude oil, achieving a sulfur reduction of 48.68 wt% with optimized parameters. In this study, UAOD surpasses ODS by achieving three times greater sulfur reduction in just 10 min, compared to 90 min of ODS.

Table 1: Different Optimization Applications using RSM

Subject of Treatment	Catalyst	Optimal Parameters	Removal Percentages	Reference
Diesel fuel containing BT and DBT	Activated Carbon Supported Phosphotungstic Acid (HPW-AC)	Mixing time = 88.5 min Mixing speed = 16,800 rpm Mixing temperature = 63.28 °C	Desulfurization rate = 43.79 % - 61.73 % Percent oxidation = 61.73 %	Barilla et al. (2022)
Gasoline and Crude Oil	Phosphotungstic Acid (PW)	Gasoline: Ultrasonic power = 400 W Irradiation time = 7 min Oxidant amount = 8 mL Crude oil: Ultrasonic power = 700 W Irradiation time = 10 min Oxidant amount = 10 mL	Gasoline desulfurization rate = 80.87 % Crude oil desulfurization rate: 73.37 %	Zhou et al. (2020)
Diesel fuel	Sodium Phosphotungstate (NaPW)	Ultrasound time = 27.1 min Amplitude = 39.9 % Catalyst dosage = 320.9 mg Temperature = 69.7 °C	Average sulfur conversion = 95.0 %	Choi et al. (2022)
Heavy Crude oil	Acetic Acid	Ultrasonic Power = 7 W/mL Catalyst amount = 4.8 wt% Oxidant amount = 2.92 wt% Reaction time = 10 min	48.68 wt%	Ghahremani et al.(2021)

5. Conclusion

This review paper comprehensively addresses the challenges associated with sulfur emissions from fossil fuels and the critical need for advanced desulfurization methods. It emphasizes the significance of UAOD and RSM in enhancing desulfurization efficiency and optimizing operational processes. The paper highlights the environmental challenges posed by sulfur emissions. It underscores the importance of regulatory standards in mitigating pollution and adverse health effects associated with sulfur compounds. The composition of sulfur in crude oil and gasoline was also analyzed, emphasizing the necessity of desulfurization processes to comply with regulatory standards and protect processing catalysts and the environment. RSM emerges as a crucial tool for optimizing desulfurization processes, offering a means to improve efficiency while minimizing costs. The paper explores UAOD as an innovative desulfurization technique, leveraging ultrasonic waves to enhance molecular interactions and promote the breakdown of sulfur compounds. It discusses the industrial applicability of UAOD and its potential to address sulfur-related challenges in fuel production. In utilizing optimization techniques, the versatility of RSM across various domains was also highlighted, showcasing its efficacy in optimizing desulfurization processes and addressing complex challenges in fuel production. Through empirical studies and recent literature, the paper underscores the pivotal role of RSM in achieving optimal operating conditions and maximizing desulfurization efficiency. In conclusion, this review paper serves as a valuable resource for researchers, industry professionals, and regulatory agencies involved in mitigating sulfur emissions from fossil fuels. By providing insights into advanced desulfurization methods and optimization techniques, it advances our understanding of modern sulfur management strategies and their implications for environmental sustainability and industrial operations.

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