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Integration of Deasphalting and Oxidative Desulfurization Processes for Heavy Fuel Oil Desulfurization

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ABSTRACT

In this study, a combined process consisting of solvent de-asphalting followed by continuous ultrasound-assisted oxidative desulfurization (UAOD) was applied for the treatment of heavy fuel oil (HFO). Acetic acid (99 wt.%) and hydrogen peroxide (35 vol.%) were employed as the catalyst and oxidant, respectively. n-Heptane and n-hexane were tested as de-asphalting solvents, while methanol and acetonitrile were used as extraction solvents in the liquid–liquid separation stage. The integrated process exhibited high efficiency in sulfur removal, reducing the sulfur content of HFO to below 5000 ppm. The most effective performance was obtained when n-heptane was used for de-asphalting, combined with ultrasonic desulfurization and subsequent extraction with methanol, which lowered the sulfur content to approximately 5292 ppm.

KEYWORDS: *De-asphalting; Ultrasonic- assisted oxidative desulfurization (UAOD); Sulfur removal; Oxidative; heavy fuel oil (HFO)*

1. Introduction

Fuel oil, as a fossil-derived energy source, exists in different grades depending on sulfur and metal content as well as physical properties. High-sulfur fuel oil (HSFO) typically contains more than 35,000 ppm of sulfur along with significant amounts of heavy metals, Conradson carbon residue, and ash, which make its upgrading particularly challenging [1, 2]. Thus, upgrading different types of fuel oils is essential. However, its upgrading is very difficult due to the high volume of pollutants. The major application of low sulfur fuel oil is the consumption as bunker fuel in the shipping industries. According to the regulation of 2020 IMO and the restrictions of the international environment, the use of fuel oil with sulfur content lower than 5000 ppm is acceptable. There are some methods for fuel oil upgrading such as fluid catalytic cracking (FCC), hydrodesulfurization (HDS), high Severity or petrochemical FCC units (HS-FCC™), different hydrocracking units such as LC-Slurry™ technology, and etc. The conventional method for the desulfurization of fuel oil and oil residue is hydrodesulfurization (HDS) at industrial scale. Sulfur-containing hydrocarbons convert to H_2S in the presence of catalysts such as Co-Mo/Al_2O_3 and Ni-Mo/Al_2O_3 at hard operating conditions (at temperature of 300–340 °C and pressure of 20–100 atm). After this process, H2S oxidized and covert to the element of sulfur in the Claus process. But HDS process has some drawbacks such as H_2S production and hard operating conditions. Moreover, this process cannot remove heterocyclic compounds such as dibenzothiophene (DBT) and benzothiophene (BT).

There are some technologies for overcoming these challenges. One of the main technologies is oxidative desulfurization process (ODS) that is promising due to its operation at ambient temperature and pressure. Moreover, this process has high potential in removal of aromatic sulfur compounds that are not reactive in HDS [3-7]. It removes sulfur compounds during the two steps; the formation of sulfones from sulfur compounds and, the elimination of sulfones and sulfoxides from fuel. Usually, liquid-liquid extraction is

applied to separate sulfones [8]. Moreover, ultrasound assisted oxidative desulfurization (UAOD) as an improved ODS method has been noticeable in recent years [9]. Recent studies have focused on improving oxidative desulfurization through catalyst development and process intensification. For instance, Albayrak and Tavman (2022) [9] provided a comprehensive review of sono-oxidative desulfurization strategies. More recently, Barilla et al. (2023) [1] reported the use of activated carbon-supported phosphotungstic acid under ultrasonication for deep desulfurization. In parallel, novel approaches for solvent de-asphalting have been explored; Saha et al. (2023) [10] demonstrated microwave-assisted SDA of HFO, achieving high asphaltene removal efficiency. Furthermore, Sadeghi and Shahhosseini (2024) [11] investigated the combined application of de-asphalting and oxidative desulfurization for HFO, showing the potential of integrated methods.

Despite these advances, the majority of studies either focus on light fuels (diesel, kerosene, gasoline) or investigate SDA and ODS/UAOD as separate processes. To date, very limited research has addressed their integration in a continuous system for the desulfurization of heavy fuel oil. This gap highlights the need for exploring combined SDA–UAOD processes, which can simultaneously mitigate the negative impact of asphaltenes and achieve regulatory sulfur levels for marine fuel applications.

The ultrasonic reactor leads to lower operating costs, higher safety and environmental protection. In this process, the use of ultrasonic reactor system enhances the desulfurization rate due to a highly effective dispersion and thus improved reaction kinetics. Ultrasonic process increases mass transfer between organic and aqueous phases in the reaction under nano-scale dispersions. Ultrasonic cavitation enhances the mass transfer and reaction rate by extreme conditions. During the cavitation bubble implosion, very high temperatures (~ 5,000K) and pressure (~ 2,000 atm) are locally obtained. Moreover, the implosion of the cavitation bubble leads to the formation of liquid jets with high velocity, which creates very high shear forces. These extraordinary mechanical forces decrease the time of oxidation reaction and increase the sulfur conversion efficiency within seconds. Ultrasonic oxidative desulphurization is highly effective for the removal of hardly removable sulfur refractory compounds (e.g., 4,6-dimethyldibenzothiophene and other alkyl-substituted thiophene derivatives) [9, 12]. In this process, oxidizer and catalyst is the main consumption materials. It should be noted that the catalyst can be recovered in the next steps and be reused due to environmental issues. And therefore, it is not a consumable material. In this case, it is necessary to design a catalyst recovery unit [13-15]. Several studies investigated UAOD process in the presence of different catalysts [16], phase-transfer agent (PTA) [17], oxidants [4], extracting agents, and adsorbents[18] and also, investigations of the influential variables such as temperature, pressure, ultrasonic [19] and economic analysis [20] have been considered. All studies are for the desulfurization of light crude oil and their derivatives. In other words, ODS and UAOD processes cannot individually reduce sulfur content to lowest than 5000 ppm. Moreover, the energy, catalyst, and oxidizer consumption of ODS and UAOD processes be very high for fuel oil desulfurization. Therefore, it is not economical. In this study, a continuous UAOD process integrated with the solvent de-asphalting process (SDA) to reach sulfur content lower than 5000 ppm. The presence of asphaltene leads to increase fuel oil viscosity which creates different challenges for burning in the engine and also it forms coke. However, some technologies such as HDS and FCC decrease asphaltenes. But the presence of asphaltene content of fuels causes to deactivation of catalysts due to coke formation, plugging, and fouling of lines and reactors. SDA is a usual method for the asphaltenes separation from HFO. Saha et al. [10] investigated de-asphalting process for upgrading fuel oil. They used from thee solvent n-hexane, n-heptane, and n-pentane for HFO deasphalting by ultrasound-assisted, microwave-assisted, and supercritical solvent deasphalting methods. The results showed the upper than 80 wt.% asphaltenes from HFO using heptane and hexane. Sadeghi & Shahhosseini [11] investigated the ODS process HFO by hydrogen peroxide and acetic acid as oxidant and catalyst, respectively. They used dimethylformamide (DMF) as an extractive solvent in the liquid-liquid extraction. The showed that the use of oxidation and extraction can lead to removing HFO sulfur content to 0.91 wt%.

Unlike previous studies that primarily investigated ODS and UAOD for light fractions of crude oil such as diesel, kerosene, and naphtha, the present work focuses on the desulfurization of heavy fuel oil, which contains much higher

levels of sulfur and asphaltenes. To the best of our knowledge, this is the first study to demonstrate the effectiveness of combining solvent de-asphalting (SDA) with continuous UAOD for HFO upgrading. This integrated approach not only addresses the limitations of ODS/UAOD when applied individually but also achieves sulfur reduction to below 5000 ppm, making the process relevant for meeting IMO 2020 marine fuel standards.

2. Materials and Methods

2.1. Materials

Hydrogen peroxide (H_2O_2: 35 vol%) were purchased from Merck Inc. Acetic acid (99 wt.%) was bought from Merck Inc. Solvent de-asphalting including n-heptane and n-hexane (98%) were obtained from Merck Co. Methanol and acetonitrile were procured from Sigma-Aldrich. The heavy fuel oil (HFO) was used as fuel from a commercially available heavy fuel oil.

2.2. Experimental method

The experiments were done by preparing commercial HFO as feed under ultrasonic irradiation in a jacket type glass reactor (Fig. 1). The feed specifications are shown in Table 1. The reactor was equipped with a circular water bath through the reactor jacket to adjust constant temperature due to the nature of the exothermic reaction and the thermometer to display temperature. The used model of UAOD was UTD 400, Ultrasound Technology Development Company, Iran. All reactions were done at 20 kHz frequency. The laboratory steps include two steps including de-asphalting process and the desulfurization process.

The first step includes mixing HFO with n-heptane or hexane as solvent of de-asphalting at the ratio of 2:1 (v/v). The stirring was done for 2 hours at ambient temperature to ensure homogeneous contact; and then solvent de-asphalting processes resulted in precipitates of asphaltenes at 4h. The precipitates were filtered by silica gel and dried at 100 °C. The de-asphalted HFO was obtained by diluent elimination from the solution by rotary evaporation. Typical mass balance per run: 100 mL HFO mixed with 50 mL solvent (yielding ≈150 mL mixture); the reported de-asphalted fraction used in subsequent UAOD experiments was 50 mL per run.

For each UAOD run, 50 mL of de-asphalted HFO (obtained as above) was diluted with 50 mL toluene (1:1 v/v) to reduce viscosity and allow probe immersion. Ultrasonic irradiation was applied at 20 kHz and 150 W power. The reactor content was heated and maintained at 40 °C. The oxidant and catalyst were added dropwise: acetic acid (99 wt.%), 2 mL and hydrogen peroxide (35 vol%), 5 mL per 50 mL de-asphalted HFO (i.e., catalyst ≈4% v/v and oxidant ≈10% v/v relative to de-asphalted HFO). The ultrasonic treatment duration was 30 minutes (selected based on preliminary tests and literature on UAOD for similar matrices). After the reaction, the two phases were separated. The prepared samples showed in Table 2.

The organic phase (hydrocarbon) was subjected to polar solvent extraction to remove oxidized sulfur species: 30 mL of the hydrocarbon phase was mixed with 30 mL of extraction solvent (methanol or acetonitrile) and stirred for 30 min. Phases were separated by decantation; the hydrocarbon phase was then washed with deionized water separated again.

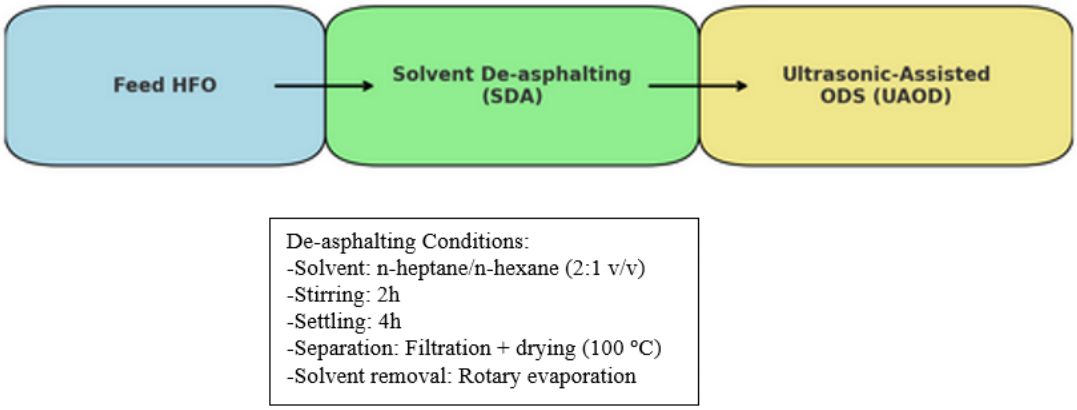

Fig. 1. Schematic of used ultrasonic system in this study.

Table 1. Used feed specification

Row	Property	Unit	Test Method	Typical value
1	Density@15 ˚C	Kg/m^3	ASTM D1298	947.9
2	Kinematic Viscosity@50 °C	cst	ASTM D445	371.5
3	Pour point	°C	ASTM D97	-12
4	Flash point	°C	ASTM D93B	53
5	Sulfur content	wt.%	ASTM D4294	2.57
6	Zinc	ppm wt	IP-470	1
7	Nickel	ppm wt	IP-470	16
8	Sodium	ppm wt	IP-470	18
9	Calcium	ppm wt	IP-470	5
10	Aluminum	ppm wt	IP-470	6
11	Silicon	ppm wt	IP-470	13
12	Vanadium	ppm wt	IP-470	157

Table 2. Prepared samples in this study.

Sample	Process	De-asphaltene solvent	extraction solvent
1	De-asphalting and ultrasonic	Hexane	Acetonitrile
2	Ultrasonic	-	Acetonitrile
3	Ultrasonic	-	Methanol
4	De-asphalting and ultrasonic	Heptane	Methanol

3. Characterizations

The EDXRF Sulfur Analyzer RX-360SH was used to measure the total sulfur content of the samples. Energy dispersive X-ray fluorescence technique was applied in this test according to the ASTM D4294. Moreover, fuel characterization such as density, kinematic viscosity, pour point, flash point, sulfur content, zinc, nickel, sodium, calcium, aluminum, silicon, vanadium was measured by ASTM D1298, ASTM D445, ASTM D97, ASTM D93B, ASTM D4294 IP-470.

Fourier transform infrared spectroscopy (FTIR) was used to confirm fabrication sulfones and sulfoxides by Bruker spectrometer (TENSOR 27) in the range of 500 to 4000 cm^{-1} at the resolution of 1 cm^{-1} for each spectrum.

4. Results and discussion

FTIR analysis of samples 1 and sample 4 before the extraction step by non-polar solvent is shown in Fig. 2 and Fig. 3, respectively. Moreover, the FTIR analysis of the feed is shown in Fig. 4.

FTIR spectra of the feed and oxidized samples were recorded to identify oxidative products. After oxidation, new absorption bands and/or an increase in intensity were observed in the region 1000–1350 cm^{-1}, which correspond to S=O stretching modes of sulfoxides and sulfones. Specifically, the appearance/enhancement of bands at ~1300–1350 cm^{-1} (asymmetric $\nu as(SO_2)$) together with bands at ~1120–1150 cm^{-1} (symmetric $\nu s(SO_2)$) is indicative of sulfone formation, while features in the ~1030–1070 cm^{-1} range are consistent with sulfoxide species. The band at ~1718 cm^{-1}, which increases in intensity in the oxidized samples, is assigned to C=O stretching of carboxylic acids (or residual acetic acid) formed as secondary oxidation products. FTIR spectrum of Sample 1 (Fig. 2) measured before extraction. Peaks at ~1300 and ~1120 cm^{-1} correspond to νas and νs

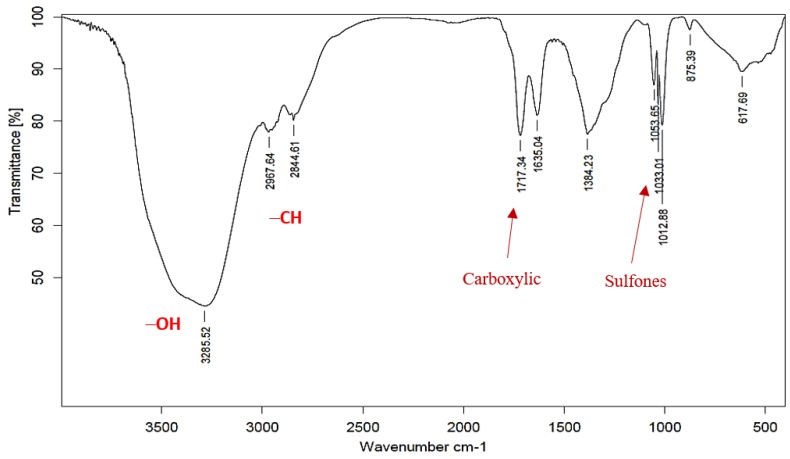

Fig. 2. FTIR spectra of samples 1 before extraction step by polar solvent.

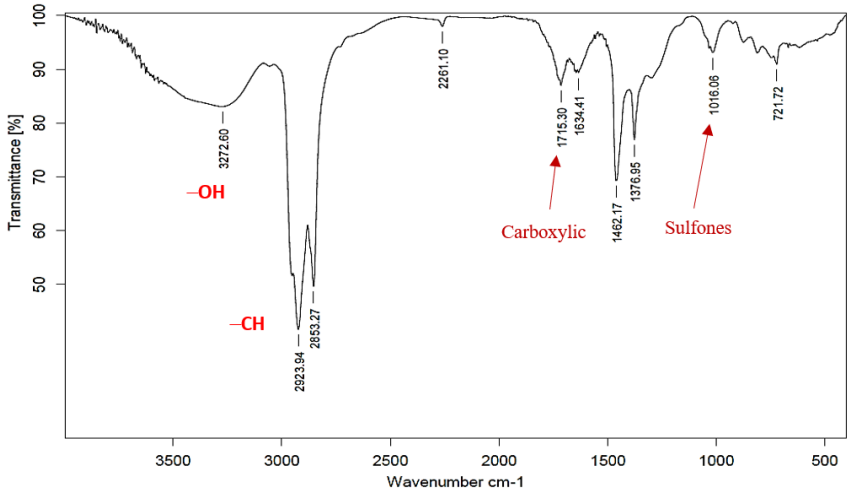

Fig. 3. FTIR spectra of sample 4 before extraction step by polar solvent.

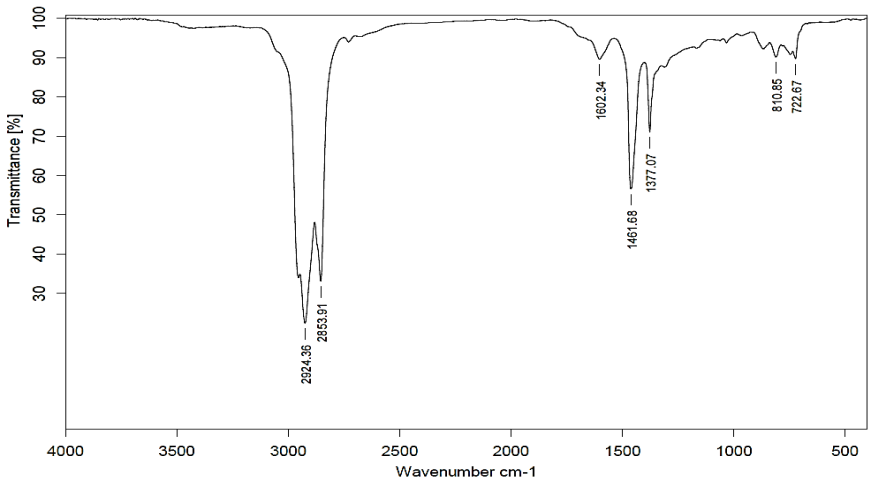

Fig. 4. FTIR spectra of use feed in this study.

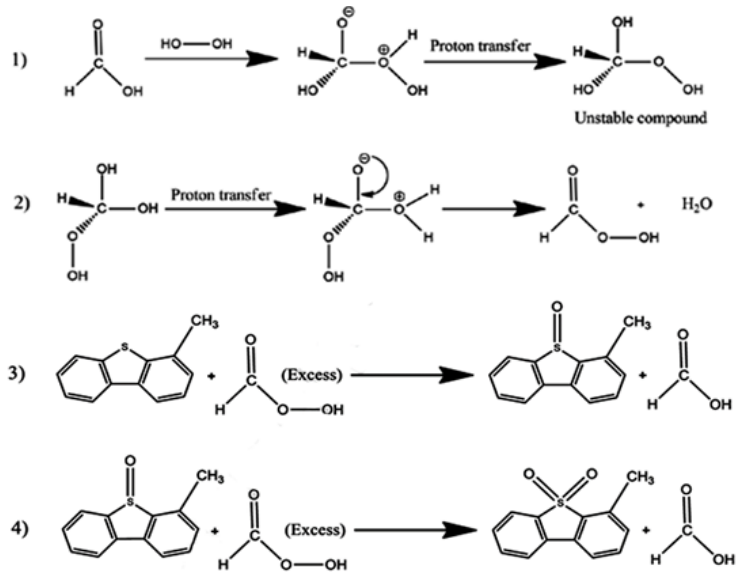

Fig. 5. Mechanism reactions between formic acid/acetic acid and hydrocarbons.

modes of sulfones, respectively; the band near 1718 cm^{-1} indicates formation of carboxylic acids. FTIR spectrum of Sample 4 (Fig. 3) measured before extraction. The enhanced absorption in the 1000–1350 cm^{-1} region confirms formation of oxidized sulfur species (sulfoxides/sulfones). Characteristic aliphatic C–H stretching (~2850–2950 cm^{-1}) and aromatic C=C bands (~1600 cm^{-1}) are visible in the Fig. 4 for FTIR spectrum of the feed. Comparison with oxidized samples demonstrates the emergence of S=O bands after treatment.

According to the experimental methods, in the reaction between catalyst and oxidizer, hydrogen peroxide converts to free radicals. The reactions in the absence of the PTC is shown in Fig. 5. This reaction includes 2 steps. First, peroxyformic acid as an unstable compound produced in the result of the reaction of hydrogen peroxide and formic acid/acetic acid in the aqueous phase. Second, peroxyformic acid is transferred to the organic phase and S-containing compounds such as dibenzothiophene convert to sulfones and sulfoxides [2, 4, 17].

The effect of temperature on the ultrasound oxidation reaction in the range of 20 to 90 °C has been studied in different published studies. The maximum boiling point of used materials is 90 °C. In other words, the used materials are volatile. Therefore, there are two limitations to temperature increasing: the decomposition of hydrogen peroxide in high temperature, and the light components evaporation of naphtha. In this study, de-asphalting was first done to remove asphalts and metals. After that, de-asphalted HFO was desulfurized by the ultrasonic reactor. The comparison between the used process for desulfurization was considered in this study. As shown in Fig. 6, sample 4 with initial sulfur content of the feed heavy fuel oil was 2.57 wt.% (≈25,700 ppm), including both processes of de-asphalting with heptane as solvent and ultrasonic, and then solvent extraction with methanol has the best result with sulfur content of 5292 ppm corresponding to a sulfur removal efficiency of approximately 79.4%. This level of removal is consistent with other reported values for heavy fractions. For instance, Sadeghi & Shahhosseini (2024) [11] achieved ~0.91 wt% sulfur after oxidative desulfurization of HFO combined with extraction, while Rahimi et al. (2017) [3] reported >90% removal in light diesel feeds. The relatively lower efficiency in our case can be attributed to the complex composition of heavy fuel oil, which contains asphaltenes, metals, and other refractory components that hinder desulfurization. With respect to IMO 2020 regulations (<0.5 wt% sulfur), the achieved result of 0.529 wt% is slightly above the limit. This shows that the proposed integrated process is capable of significantly reducing sulfur content and approaching the IMO standard, although further optimization (e.g., adjustment of oxidant/catalyst dosage, repetition of the UAOD step, or integration with additional upgrading technologies) will be necessary to fully meet the regulatory requirement.

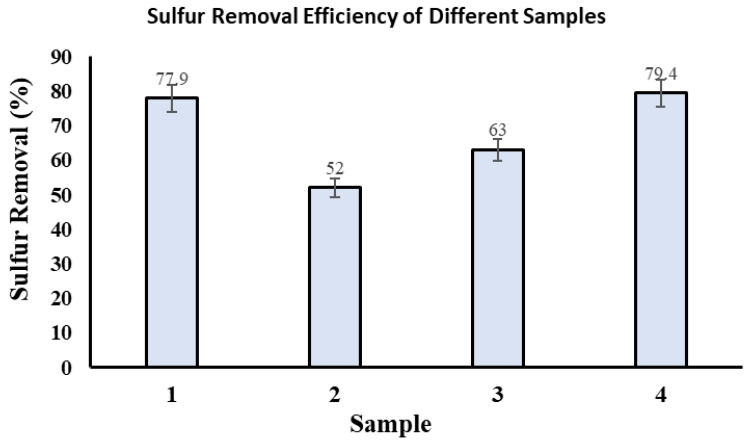

Fig. 6. Sulfur content of prepared samples.

Table 3. Summary of presents the final sulfur content, sulfur removal efficiency, and the main operating conditions.

Sample	Process Description	De-asphalting Solvent	Extraction Solvent	Final Sulfur Content (ppm)	Removal Efficiency (%)	Main Operating Conditions
1	SDA + UAOD	Hexane	Acetonitrile	~8,100	~68.5	150 W, 40 °C, H_2O_2 + AcOH
2	UAOD only	–	Acetonitrile	~12,400	~51.7	150 W, 40 °C, H_2O_2 + AcOH
3	UAOD only	–	Methanol	~10,300	~59.9	150 W, 40 °C, H_2O_2 + AcOH
4	SDA + UAOD	Heptane	Methanol	~5,292	~79.4	150 W, 40 °C, H_2O_2 + AcOH

Oxidative desulfurization technology using an ultrasonic reactor is one of the technologies of effective and compatible with the environment for desulfurization. In recent years, the use of the desulfurization method of fuel materials such as diesel, kerosene, and gasoline has been comprehensively investigated. But all these progress related to light crude oil and upgrading of heavy oil residue, fuel oil such as bunker fuels, was not considered in the different research. Therefore, it is essential to more investigations of heavier streams by ODS and UAOD process, and obtain high-value of clean fuels. The ultrasound waves increase the dispersion of carbon, water and oil phase, promote the interfacial mass transfer, and this leads to accelerating the reaction. The ultrasound waves did not affect the chemical or physical properties of the fuel. The chemical analysis of treated fuel oil showed that <1% of the hydrocarbon fuel compounds were oxidized in the ODS process. Moreover, it was found that the hydrogen peroxide amounts lead to increase oxidation rates of sulfur compounds and thus the improvement of desulphurization efficiency [21]. Table 3 shows summary of presents the final sulfur content, sulfur removal efficiency, and the main operating conditions for each experimental sample.

Although the present work was carried out at laboratory scale, the results provide insights into the potential industrial application of the integrated SDA–UAOD process. Compared to conventional hydrodesulfurization (HDS), the proposed method operates under mild conditions (ambient pressure, <100 °C), which significantly reduces energy costs. The main contributors to process cost are hydrogen peroxide and organic solvents. However, solvent recovery and recycling systems are well-established in industry and could reduce operating expenses. Moreover, solvent de-asphalting (SDA) is already applied at industrial scale, suggesting that coupling it with UAOD would be technically feasible.

From a scalability perspective, ultrasonic

reactors can be implemented in continuous-flow configurations, and previous studies have demonstrated their successful operation at pilot scale for light fuels. The integration of SDA and UAOD therefore represents a promising route for upgrading heavy fuel oils to meet IMO sulfur specifications (<5000 ppm), while potentially offering a more cost-effective and environmentally friendly alternative to conventional HDS. Further techno-economic evaluation and pilot-scale testing are recommended to validate the process feasibility.

5. Conclusion

In this study, solvent de-asphalting process and then continuous ultrasonic desulfurization were applied to desulfurization of heavy fuel oil (HFO) by acetic acid and hydrogen peroxide as catalyst and oxidant, respectively. Different solvents such as n-heptane, n-hexane were used as a solvent of the de-asphalting process. Methanol and acetonitrile were applied as an extractive solvent in the liquid-liquid extraction. The results showed the high capacity of integrated processes for desulfurization of HFO to less than 5000 ppm. Considering that there are not many studies on fuel oil desulfurization and many studies in this field have reached a dead end, the present work can guide future studies in the direction of integrating other processes with desulfurization processes and be useful for future studies.

A limitation of the present study is that each experimental condition was tested once, without replicate runs. Consequently, statistical measures such as standard deviations or error bars could not be reported. While the results demonstrate the feasibility of the integrated SDA+UAOD process, future work will focus on conducting repeated experiments and applying statistical analysis to assess reproducibility and provide confidence intervals for the measured sulfur removal efficiencies. Another limitation of the present study is the lack of kinetic measurements and time-resolved sulfur concentration data. Only overall sulfur removal efficiencies are reported (best case: 79.4% removal, from ≈25,700 ppm to 5292 ppm). To fully understand the reaction mechanism and to optimize the process, future work will include time-course experiments, extraction of apparent rate constants (k_app), mass balances on sulfur between organic/aqueous/solid phases, and determination of activation energy via Arrhenius analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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