



Research Article

Ultrasound-assisted desulfurization of crude oil using sodium hydroxide

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ABSTRACT

To meet the required standards on air quality and emissions from engine exhaust, several countries have instituted policies to set and reduce the limit of crude oil and petroleum product sulfur concentration to very low levels. This research studies the effectiveness of combining the assisted ultrasound desulfurization, and the traditional sodium hydroxide desulfurization methodologies, to lower the amount of sulfur contained in fuels produced even further. The sample in question was 25mL of crude oil obtained from the East Baghdad Oil Field in Iraq, containing 4.31 wt.% sulfur. The main underlying parameters of the UADS process; sodium hydroxide concentration, (temperature, 20-80 C°), reaction time (10-30 min), and intensity (30-60%) of ultrasound. The study contrasted the ultrasound desulfurization of crude oil performing the sodium hydroxide process with 'standard' caustic desulfurization, using the same parameters. The synergy of NaOH and ultrasonic irradiation in desulfurization of crude oil has surpassed their individual capabilities. The experimental data also compares the ultrasonic assisted empirical and reaction measures of the crude oil deuterium capture. She concluded that the maximal sulfur concentration deduced from the system was close to 68% at optimal conditions of 3M NaOH concentration, reaction temperature 60 C°, 30 minutes, with 40% of collimated ultrasound.

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INTRODUCTION

Sulfur compounds in crude oil pose significant environmental challenges due to their detrimental effects, including catalyst deactivation and corrosion in petroleum production, processing, and pipeline systems [1]. These compounds can be classified into two main categories based on their chemical behavior: active and inactive sulfur. Active sulfur compounds—such as hydrogen sulfide, mercaptans, and elemental sulfur—can directly interact with

metallic surfaces, leading to equipment corrosion. On the other hand, inactive sulfur compounds, including sulfides, disulfides, and thiophenes, do not react directly with metals but can generate hydrogen sulfide when heated, owing to their poor thermal stability [2]. Elevated sulfur content in crude oil often results in similarly high sulfur levels in refined petroleum products. Regulation of sulfur content is required because the compound is highly toxic, corrosive in nature, and has an ecological impact. The main two acid rain causes are the sulfur release and the nitrogen oxides.

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Thus, for avoiding such unsafe ecological consequences, desulfurization processes have been used widely [3, 4]. Although it has lesser applications in crude oil, catalytic hydrodesulfurization (HDS) is the most widely practiced desulfurization process in refinery operations for petroleum fractions. HDS is effective for the removal of active sulfur species like mercaptans and hydrogen sulfide, but not for disulfides. Sulfur disulfides are resistant to HDS processing and are transported through in the treated crude due to their low reactivity and high thermal stability [5, 6]. High temperatures (300–350°C) and pressures (15–90 bar) are utilized for the HDS process to operate at its best. It also contains costly cobalt-molybdenum catalysts and a substantial amount of hydrogen [7–10]. All these disadvantages have encouraged scientists to search for cheaper and efficient alternative methods for the desulfurization of fuels like oxidative desulfurization, adsorptive desulfurization, extraction desulfurization, alkylation desulfurization, bio-desulfurization, radiation desulfurization, and caustic desulfurization using sodium hydroxide (NaOH) [11–14]. Sodium hydroxide is a strong base that still remains a good option for desulfurization due to its simplicity, low price, and effectiveness in removing a high amount of sulfur compounds from sour crude oil [15].

In 1993, Ahonen [16] employed an electrolysis cell to oxidize and remove sulfur from heavy oil fractions blended with a sodium hydroxide solution. This process resulted in the conversion of most sulfur compounds into sulfites or sulfates. K. Jeyajothi [17] evaluated the effectiveness of sulfide removal from crude oil using the caustic washing method with varying NaOH concentrations. The desulfurization efficiency exhibited a notable reduction in H₂S content, achieving 50% efficiency at a concentration of 0.2 N NaOH. Shakirullah et al. [10] investigated the application of sodium hydroxide for the desulfurization of various petroleum products, including diesel oil, heavy residue, kerosene, and commercial furnace oil, and observed that the desulfurization efficiency varied among products. Specifically, kerosene exhibited an efficiency of 60%, diesel oil 68%, heavy residue 70%, and commercial furnace oil 71%. Al-Khodor and Albayati [15] utilized sodium hydroxide to remove sulfur from high-sulfur crude oil containing 5.8% sulfur by weight. Following theoretical optimization and experimental evaluation, a desulfurization efficiency of 56.89% was achieved. Kumari and Sengupta [18] employed ultrasound oxidation to investigate the simultaneous denitrogenation and desulfurization of model fuel and found that ultrasound improved oxidation efficiency and enhanced the conversion of sulfur-based compounds up to 93.7%.

Ultrasound consists of pressure waves that pulsate at extremely higher frequencies than sound waves that the human ear can hear, which ranges between 20 and 20,000 Hz [19]. A gas, liquid, or solid medium can produce waves when it undergoes a shock or vibratory motion displacement of a particle or a number of particles which can be

either singular or repetitively triggered. A remarkably high, linear power density of sound waves leads to a phenomenon known as ultrasonic waves. This phenomenon involves the formation and “violent” collapse of gas bubbles which subsequently creates shock waves that speed up chemical reactions and increase their effectiveness. The fleeting collapse of gas bubbles produce high fluid waves and the pressure and temperature raised to 1000 atm and 5000 K, respectively, over the points of explosions. The very high, center local conditions in an explosive manner foster rapid chemical reactions of the system by forming active intermediates in the micro environment [20].

Different strategies for desulfurization have had some success, but have also struggled with efficiencies targeting complex sulfur species and high energy consumption. This study focuses on ultrasound technology to overcome these hurdles by integrating ultrasound with caustic desulfurization. Ultrasound-assisted desulfurization (UADS) is able to use acoustic ‘cavitation’ to promote and enhance sulfur compound breakdown, and improve mass transfer over time. The combination of ultrasound and caustic soda is expected to improve the rate of desulfurization, while also reducing temperature and pressure operational conditions. This results in lowering energy demands and improving process efficiencies. The resultant new method is also expected to process the material in less time, leading to higher throughputs and productivity. UADS in particular helps target complex sulfur compounds, something traditional methods find very difficult. Also, the use of ultrasound in UADS over the traditional methods helps ensure compliance with international agreements by reducing sulfur emission, and supporting cleaner fuel production with less overall desulfurization energy consumption.

This work aims to confirm the effectiveness of UADS in reducing the sulphur content of crude oil to below 50 ppm using NaOH, as well as to assess the effect of fundamental UAD processing parameters—NaOH concentration, temperature, time, and ultrasound power—on the process. By addressing these parameters, this research aims to close existing gaps in the desulfurization process and establish a scalable, energy-efficient solution for producing cleaner fuels.

MATERIALS AND METHODS

Materials

In all experiments, crude oil samples were obtained from the East Baghdad Oil Field in Iraq. The sodium hydroxide (NaOH) used had a purity of 95%. For washing the glassware, 99.5% pure toluene (C₆H₅CH₃) was employed. All chemicals used were commercial laboratory supplies purchased from local suppliers and were utilized without prior preparation or purification. The physical parameters of the sample are detailed in Table 1.

Table 1. Properties of East Baghdad Crude Oil

Property	Value	Unit
API gravity	21.8	-
Specific gravity SG	0.923	-(at 15.56 °C)
Kinematic viscosity	78.731	cSt (at 26.7 °C)
Sulfur content	4.31	wt. %
H ₂ S content	4.36	ppm
Sediment content	0.0126	vol. %
Micro carbon residue	9.947	wt. %
Asphalting	6.412	wt. %
Salt content	21	ppm
Water content	1.60	wt. %

Equipment

The ultrasonic experiments were conducted using a Digital Ultrasonic Cleaner, a flexible and powerful ultrasonic liquid handler from Guangzhou CinTuo Technology Co., Ltd., China. The program-controlled microprocessor-based equipment was utilized at 800 watts and 40 kHz with 10 to 30 minutes of time settings and an 80 °C maximum temperature. For routine non-ultrasonic tests, the work was done with the help of a 2000 rpm mechanical stirrer to obtain effective mixing. The experiments were carried out with a 150-mL Pyrex Griffin beaker. Upon ultrasonic and non-ultrasonic treatment, a 100-mL cone-type glass separating funnel was used in the solvent

extraction process. The total sulfur content of crude oil was determined by RX-630SA (Cannon Instrument Company, USA), which uses energy-dispersive X-ray fluorescence (EDXRF) according to ASTM D4294-03 and ISO 8754 standards. This method is renowned for being quick, not destructive, cost-effective, and accurate.

Experiments

To perform the desulfurization process, 25 mL of crude oil is first placed in a beaker, and 15 mL of NaOH solution with varying concentrations (ranging from 1M to 4M) is added. NaOH powder is dissolved in distilled water to prepare the NaOH solution. The contents of the beaker are sonicated with continuous stirring to facilitate good mixing. The beaker is then immersed in distilled water and loaded into an ultrasonic processor. Sonication is activated with an ultrasonic generator whose control is preset at the desired reaction temperature. After the pre-programmed sonication time has elapsed, the ultrasonic generator is turned off, and the reaction mixture is transferred into a separator funnel. The funnel is hand-shaken for 2 minutes to allow phase separation and left standing for 10 minutes to separate the organic phase from the aqueous phase distinctly. A small sample from the organic phase is collected to test the sulfur concentration. Detailed experimental setup for the traditional NaOH desulfurization process can be found in previous research [15]. the same procedure is followed in non-ultrasonic experiments, with the ultrasonic apparatus being replaced by the mechanical stirrer. The experimental process for UADS is illustrated in Figure 1, while Table 2 details the experimental conditions and procedures..

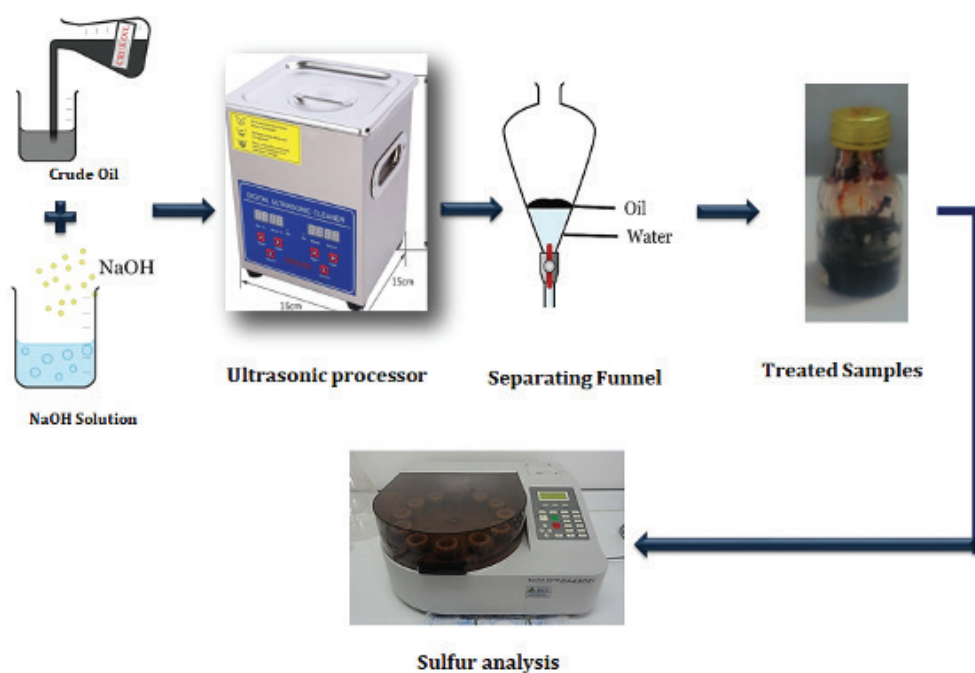
**Figure 1.** The UADS experimental setup.

Table 2. Specifications of experimental operations

Exp. Number	Parameter	Variable	Operating Conditions	Ultrasonic Tests	Non- Ultrasonic Tests
1		1			
2		1.5			
3		2			
4	Molarity of NaOH	2.5	time = 30 min; temp = 60 °C	frequency = 40KHz	Speed = 2000 rpm
5		3			
6		3.5			
7		4			
8		20			
9	Temperature in Celsius	40	time = 10 min; NaOH = 3M	frequency = 40KHz	Speed = 2000 rpm
10		60			
11		80			
12		20			
13	Temperature in Celsius	40	time = 20 min; NaOH = 3M	frequency = 40KHz	Speed = 2000 rpm
14		60			
15		80			
16		20			
17	Temperature in Celsius	40	time = 25 min; NaOH = 3M	frequency = 40KHz	Speed = 2000 rpm
18		60			
19		80			
20		20			
21	Temperature in Celsius	40	time = 30 min; NaOH = 3M	frequency = 40KHz	Speed = 2000 rpm
22		60			
23		80			
24		30			
25	Ultrasound amplitude %	40	time= 30 min; temp = 60 °C;		
26		50	NaOH = 3M		
27		60			

Sulfur Removal Efficiency (η %)

Desulfurization efficiency is calculated using the following equation: it is the ratio of the sulfur removed to the sulfur initially present in the crude oil. The desulfurization efficiency (η) is determined using the equation:

$$\eta (\%) = \left(\frac{C^o - C}{C^o} \right) \times 100 \quad (1)$$

Where C^o is the original sulfur concentration (ppm), and C is the sulfur concentration in the treated sample (ppm).

RESULTS AND DISCUSSION**Comparison between Ultrasonic and Conventional Desulfurization using NaOH**

The study investigates the comparative effect of ultrasonic-assisted desulfurization and traditional NaOH-based

desulfurization procedures, respectively, through performance comparisons at different NaOH concentrations, temperatures, and reaction times. Figures 2, 3, and 4 indicate that ultrasonic desulfurization tends to outdo plain NaOH treatment alone in rates of sulfur elimination. Internal benefit of ultrasonic treatment is the physical phenomenon of cavitation, where the high-frequency sound waves produce tiny bubbles that collapse at a rapid rate. The collapse creates infinitesimal areas of high pressure and temperature, and this increases the rate of desulfurization tremendously. These intensification effects of ultrasound, including enhanced mass transfer and reaction kinetics, are consistent with findings reported in other chemical processes [21]. It does so by speeding up the reaction between sulfur in the crude oil and sodium hydroxide (NaOH) and enhancing mass transfer. Ultrasonic waves provide the necessary activation energy sulfur removal and is at a much lower energy barrier, hence making the process much faster and more efficient than with normal techniques.

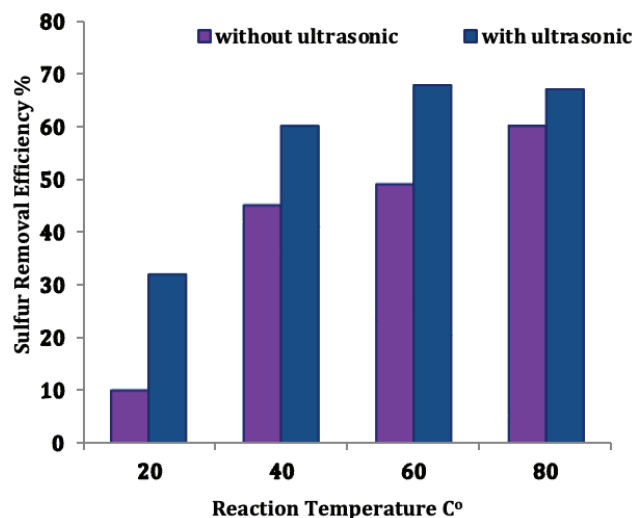


Figure 2. Ultrasonic desulfurization versus traditional NaOH desulfurization at different temperatures, 30 minutes, and 3M NaOH concentration.

Additionally, the mechanical action of cavitation that also decomposes sulfur compounds increases the active surface area of the NaOH-sulfur reaction and enables more efficient interaction of NaOH with sulfur [19]. These synergistic effects—fast reaction kinetics, mass transfer optimum, and accessibility of sulfur sites—lead to the greater result of desulfurization by ultrasound.

Figure 2 shows the effect of temperature on desulfurization efficiency with and without ultrasonic waves. The most striking enhancements are found at low to mild

temperatures (20°C to 60°C). With 20°C, ultrasonic support significantly enhances efficiency with a considerable performance compared to traditional methods. With 60°C, the rate of sulfur elimination is optimal at around 70%, with the best condition for ultrasonic desulfurization. However, at a temperature increase to 80°C, the advantages of ultrasonic assistance diminish, suggesting that at some sort of thermal ceiling, there is little to be gained by further energy from the ultrasonic source. The pattern is such that synergism between ultrasound and temperature is best at lower temperatures and that this is likely due to more extensive cavitation at these conditions, which causes maximum mass transfer and reaction rate.

Figure 3 illustrates the effect of reaction time on desulfurization efficiency. Without the use of ultrasonic assistance, sulfur removal increases incrementally from 10% efficiency after 10 minutes and eventually 50% after 30 minutes. With the use of ultrasonic assistance, the desulfurization process is significantly improved. For the first 10 minutes, the efficiency of sulfur removal is already at about 30%, while it is about 70% at 30 minutes. This rapid jump in efficiency upon ultrasonic treatment indicates the enhanced reaction kinetics with cavitation. Cavitation effect will improve the mixing and mass transfer so that NaOH will be more reactive with sulfur compounds within a shorter time frame and thus achieve maximum desulfurization within a shorter time period.

Figure 4 shows the effect of NaOH molarity on sulfur removal efficiency with or without the assistance of ultrasonic. As seen from the curve, as there is a transition in the quantity of NaOH from 1M to 3M, there is therefore an increase in sulfur removal efficiency. It is due to the fact that greater NaOH will be present to react with sulfur

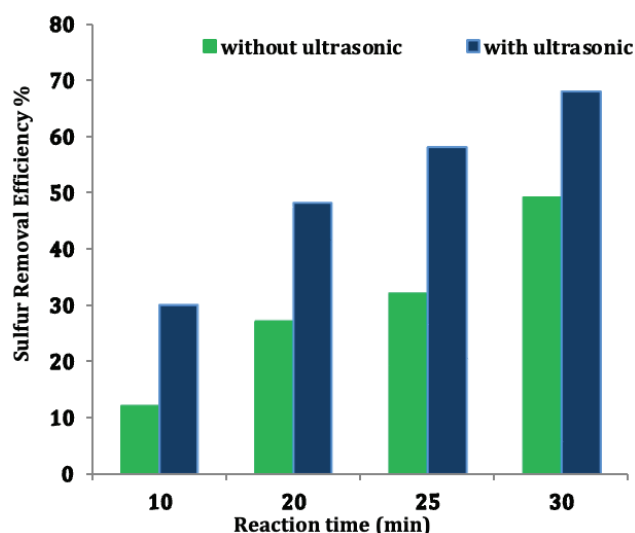


Figure 3. Comparison of ultrasonic desulfurization versus traditional NaOH desulfurization at different time intervals, operating at a temperature of 60°C with a NaOH concentration of 3M.

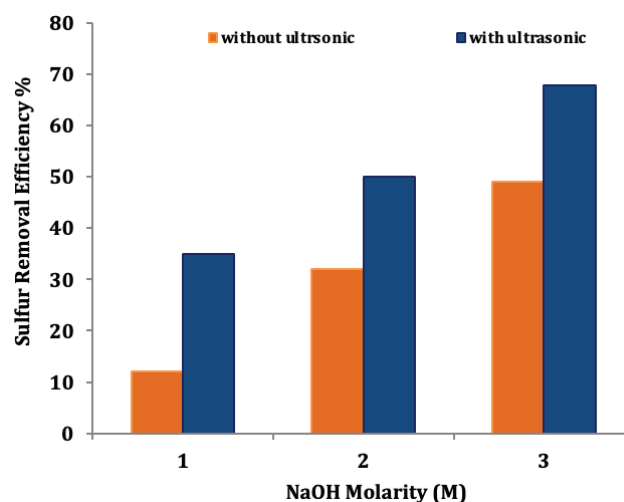


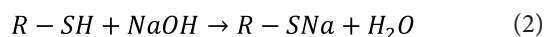
Figure 4. Comparison of ultrasonic desulfurization versus traditional NaOH desulfurization at various NaOH molar concentrations, operating at a temperature of 60°C and a processing time of 30 minutes.

compounds. But by the addition of ultrasonic assistance, the impact is enhanced in all molarities, with optimal improvement at the maximum quantity of 3M, where efficiency is achieved up to 70%.

The results show not only that ultrasonic waves enhance NaOH reactivity but also optimize the utilization of higher concentrations of NaOH. This performance is likely a result of the ultrasonic waves inducing greater mixing and greater mass transfer rates, overcoming the limitations typically imposed by diffusion on typical desulfurization processes. In this regard, ultrasonic assistance becomes more active at higher NaOH concentrations and thus maximizes the opportunity for desulfurization.

Influence of NaOH Concentrations on Ultrasonic Desulfurization

Different levels of NaOH at 60°C were applied, and there was sonication under 40 kHz for 30 minutes. Figure 5 demonstrates that there was a decrease of sulfur content in the crude oil when there are higher concentrations of NaOH, indicating that the highest removal of sulfur takes place at high levels of NaOH. This is likely an outcome of the acidic components of some of the sulfur compounds, such as mercaptans, which transfer with ease from the oily phase into the alkaline sodium hydroxide layer where they are oxidized to sulfides. Since the aqueous solution of sodium hydroxide reacts with the crude oil [22], it joins forces with inorganic sulfur compounds to create salts that are eventually separated out using a separating funnel. This results in desulfurized crude oil. Equation (2) explains the reaction:



Here R-SH is the thiol (mercaptan) group, and R-SNa is the sodium salt that gets produced in the course of the reaction. This conversion allows sulfur to be removed from the oil phase because the sodium salt (R-SNa) is more soluble

in the aqueous NaOH layer and can be separated from the crude oil.

This observation is in line with previous research, where increasing the concentration of sodium hydroxide solution improves the efficiency of the desulfurization reaction [23]. Further increased concentration of NaOH to over 3 M, for instance, to 4 M, results in no better than a slight increase in efficiency of desulfurization. As the process employs a difficult-to-regenerate solvent, wasteful consumption of NaOH would not be economical. Thus, the best concentration was revealed to be a 3 M NaOH solution, which extracted sulfur with an efficiency of approximately 67.8%.

Influence of Ultrasound-Assisted Desulfurization Temperature and Time to be effect

The literature review further identifies that temperature is crucial in influencing the properties of wave propagation in sonication, and as temperatures rise there are higher sulfur conversion [24, 25]. Elevated temperatures enhance the rate of mass transfer diffusion and accelerate achieving equilibrium [26]. However, since the NaOH-sulfur compounds reaction process is exothermic, external heat is not required. Though high temperatures can accelerate the rate of reaction, extremely high temperatures may lead to negative consequences, such as desulfurization or thermal cracking of crude oil. The Figure 6 indicates the efficiency of removing sulfur (%) with temperature (°C) at different reaction times (10, 20, 25, and 30 minutes).

The first one is general trend since with the increase in temperature from 10°C to 80°C, the efficiency of sulfur removal always improves at all the reaction times, which shows that there is a positive correlation between the desulfurization efficiency and temperature. The second one is effect of reaction time for shorter reaction times (10 minutes), the efficiency of sulfur removal is very low, starting from around 10% at 20°C and only reaching around 30% at

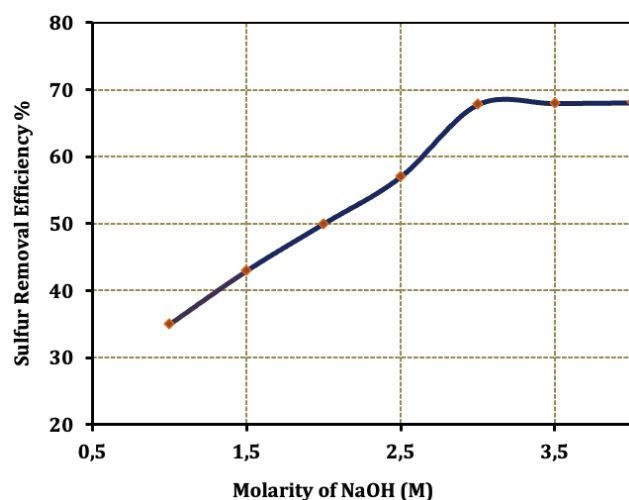


Figure 5. Influence of sodium hydroxide concentrations.

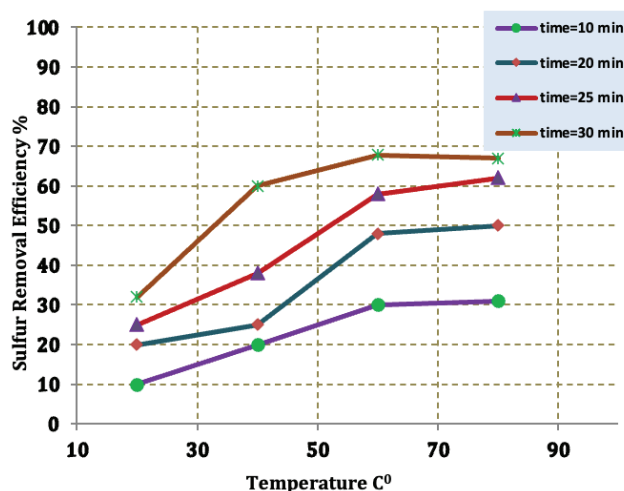


Figure 6. Influence of Ultrasound-Assisted Desulfurization Temperature and Time.

80°C. Because the reaction time is increased to 20, 25, and 30 minutes, the sulfur removal efficiency is significantly improved, especially within the range of temperatures 30°C to 60°C, with the 30-minute reaction being the most efficient at all temperatures. The optimal temperature range the sulfur removal efficiency seems to reach its peak at about 60°C for all the reaction times, especially for the 25-minute and 30-minute ranges, where it is approximately 70% and 80%, respectively. After 60°C, the improvement in efficiency plateaus or drops slightly, indicating that temperatures higher than this are not beneficial to a greater degree.

Influence of Ultra-Sonation Intensity

Since the amplitude of ultrasonic waves directly correlates with the intensity of the desulfurization process, the effect of various amplitudes (30%, 40%, 50%, and 60%) was thoroughly investigated at a fixed duration of 30 minutes and a steady temperature of 60°C. As depicted in Figure (7), the sulfur removal efficiency showed a noticeable improvement, climbing from 58% to an impressive 68% when the amplitude was increased from 30% to 40%. This striking enhancement aligns with the findings of Duarte et al., who emphasized that boosting ultrasonic intensity significantly enhances cavitation. This process generates more free radicals, activates oxygen atoms to a greater degree, and dramatically improves the overall sulfur removal efficiency [27]. Nevertheless, when the amplitude exceeded 40%, there was a slight but detectable decrease in desulfurization efficiency. This slight decrease can be explained by high-intensity ultrasound waves causing excessive bubble agitation, a phenomenon also reported in other ultrasound-assisted desulfurization studies [28]. These high waves cause excessive vibration of the bubbles during the negative acoustic phase, and thus, the bubbles fail to have enough time to collapse effectively in the positive acoustic phase [29]. Therefore, while initially increasing capacity boosts performance, exceeding 40% leads to diminishing

returns. We can consider a 40% amplitude to strike a perfect balance. This value provides just the right intensity to achieve maximum efficiency without compromising the microbubble dynamics.

CONCLUSION

In this work, the effect of combining ultrasound with sodium hydroxide treatment for crude oil desulfurization was examined. The experiments showed that ultrasound made a clear difference. The highest sulfur removal, about 68%, was achieved when a 3 M NaOH solution was used at 60 °C for 30 minutes with 40% ultrasound power. This improvement compared with the 49% obtained without ultrasound indicates that sound waves helped the caustic solution reach and react with more sulfur compounds. Even though the results were encouraging, the final sulfur content was still well above the 50 ppm level required for ultra-low-sulfur fuels. This means that the process, in its current form, is not yet sufficient for industrial use. Reaching that target will probably require extra steps, such as integrating catalytic oxidative desulfurization methods, for instance using efficient catalysts like supported ceria [30], or further tuning of the operating conditions. Still, the method shows good potential. It works under relatively mild conditions, needs less energy and fewer chemicals, and could help lower sulfur-related emissions. Future studies should test different combinations and perhaps link ultrasound with other techniques to push the sulfur level even lower and make the process more practical for cleaner-fuel production.

AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE

Artificial intelligence was not used in the preparation of the article.

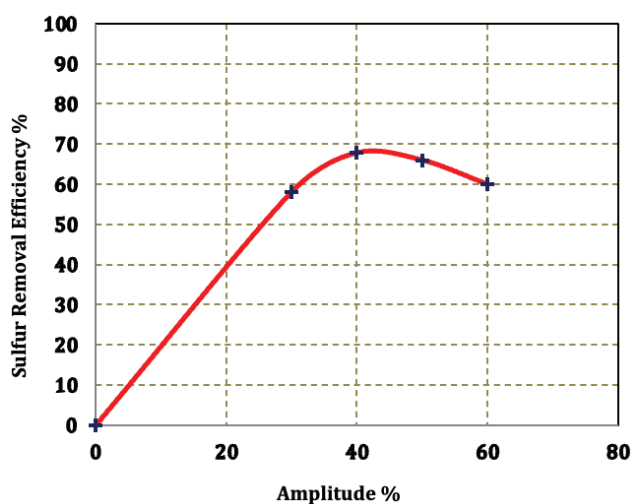


Figure 7. Influence of ultra-sonication intensity.

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