A Review on Desulfurization Techniques of Liquid Fuels

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Abstract: Strategies for liquid fuel desulfurization were estimated by reviewing desulfurization literature and critically evaluating the viability of the several methods for liquid fuels. This review paper dealt with various desulfurization technology for liquid fuels such as extractive desulfurization, oxidative desulfurization, biodesulfurization and adsorptive desulfurization to form ultra-clean liquid fuels. This work, therefore, reviews the different approaches and effect of desulfurization on liquid fuels investigating carried out on desulfurization under different process conditions.

Keywords: Oxidative desulfurization (ODS), Hydrodesulfurization(HDS), Extractive desulfurization(EDS), Biodesulfurization (BDS), Adsorptive Desulfurization, Sulfur.

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1. Introduction

Sulfur in liquid fuels is highly undesirable and the sulfur content of many products is strictly regulated. Sulfur reduces the quality of the oil needed to produce final products and by extension the commercial value of the liquid fuel. There are three major types of transportation fuels such as jet fuel, diesel oil, and gasoline which are different in composition and properties. Diesel fuel contains the most refractory sulfur compounds such as Benzothiophene, Dibenzothiophene, and4,6-dimethyldibenzothiophene to the present of sulfur content in these transportation fuels culprit's environmental pollution [1]. The combustion of refractory sulfur-containing compounds present in liquid fuels proceeds to the formation of sulfur oxide. The oxides of sulfur (SO_X) account to acid rain and atmospheric pollution, which decreases combustion efficiency fuel and enhance emission of other particles. Therefore, several countries keep stringent legislation to extent sulfur content in liquid fuel, such as in European countries sulfur(S) content <10ppm and in USA <15ppm [2]. Presently, Hydrodesulfurization(HDS) process employed to reduce sulfur content from liquid fuels but it requires high operating condition (Temperature, Pressure) and high hydrogen consumption for sulfur removal [3]. HDS process is very effective to remove aromatic sulfur compounds such as thiols, sulfides, and disulfides but it is not efficient to remove more refractory sulfur compounds are Thiophenes (Ts), benzothiophenes (BTs), dibenzothiophene (DBTs), 4,6dimethyldibenzothiophenes (4,6-DMDBTs) and their alkyl derivatives. So researchers developed alternative technology to reduce the sulfur content in liquid fuel to ultra-low as adsorptive desulfurization (ADS), oxidative desulfurization (ODS), hydrodesulfurization(HDS), biodesulfurization (BDS), extractive desulfurization(EDS), etc. The sulfur containing compounds are encountered in many areas in oil refinery and common type of refractory sulfur compounds in liquid fuels. The concentration of refractory sulfur compounds varies due to boiling point changes. The refractory sulfur compound becomes more refractory with increasing of boiling point, the dominant sulfur compounds such as thiols, thiophene, and sulfides in naphtha to

substituted benzothiophenic compounds in distillate. Recent studies on desulfurization show that sulfur content in the environment is increasing and could gesture series health consequences like the respiratory disease such as emphysema [4].

2. Desulfurization Techniques

2.1. Hydrodesulfurization (HDS)

Hydrodesulfurization process is most the widely used method in the petroleum industry to reduce sulfur content in crude oil. Usually, HDS process is operated by co-feeding of crude oil and hydrogen in a fixed bed reactor stuffed with relevant Hydrodesulfurization catalyst. At the time of conventional Hydrodesulfurization, the organic sulfur compounds primarily converted to H₂S with the reactor temperature ranges 300°C to 350°C and pressure ranges 15 to 90 bar by used of Hydrodesulfurization catalyst (NiMo/Al₂O₃, CoMo/Al₂O₃, etc.) and additional recycling of H₂S by clause process to achieve elemental sulfur [6].

HDS process is most convenient method to remove aliphatic sulfur compounds such as mercaptans(RSH), sulfides(RSR), and disulfides(RSSR) but this is not appropriate process to remove more refractory sulfur containing compounds such as thiophene, benzothiophene(DBT), 4,6-dimethyldibenzo-thiophene(4,6-DMDBT) from fuel oil [7]. In HDS process aliphatic sulfur is more reactive and removed completely from liquid fuel according to following mechanisms [4]:

Mercaptans:	R-S-H	$R-S-H+H_2$	>	$R-H+H_2S$
Sulfides:	R ₁ -S-R ₂	R_1 -S- R_2 + 2 H_2		$\mathbf{R_{1}}\text{-}\mathbf{H} + \mathbf{R_{2}}\text{-}\mathbf{H} + \mathbf{H_{2}}\mathbf{S}$
Disulfides:	R ₁ -S-S-R ₂	R_1 -S-S- R_2 + 3 H_2	→	$R_1-H + R_2-H + 2H_2S$

[8] In general the reaction mechanism of DBT through HDS process was suggested via two main routes Fig 1: one is direct desulfurization route, where sulfur removal occurs without affecting the aromatic rings and another is

desulfurization occurs via hydrogenation route, in which refractory sulfur compounds (DBT) are preferentially hydrogenated in the form of 4H or 6H-DBT and these intermediates are subsequently desulfurized. Thus, sulfur removal rate of refractory sulfur compounds achieved highest degree in hydrogenation route. peroxide(H_2O_2) mostly used one, because of its low cost, environmentally benign and active oxygen content. ODS process preferentially operated in two stages shown in the Fig 2 (i) oxidation of refractory sulfur compounds into sulfones and (ii) separation by using of extraction and adsorption method into a polar solvent [12].

Figure 1: Direct desulfurization and Hydrogenation route for Hydrodesulfurization of DBT.

Even though HDS process is still the main technology applied in the petroleum refining industry some advantages appear as it described below [9]

- a) Severe reactor conditions such as high temperature and pressure required to process more refractory sulfur compounds.
- b)Use of expensive catalyst; due to the high organometallic content of heavy hydrocarbon the catalyst life shortens as the metal (Nickel, Vanadium) sulfides causes deposit formation on the catalysts.
- c) Fouling and coking, which causes catalyst deactivation.

Wang et al. [7] studied the reactivity of refractory sulfur compounds which varies with their structure and sulfur atom environment. Low boiling sulfur containing compounds such as aliphatic refractory sulfur compounds (mercaptans, sulfides, and disulfides). The aliphatic sulfur compounds are very active in Hydrodesulfurization (HDS) process and these compounds can be easily removed from liquid fuel. The refractory sulfur compounds activities in HDS process are as follows: order (from least reactive to more reactive) 4,6-DMDBT> DBT>BT>thiophene.

2.2. Oxidative Desulfurization (ODS)

The deep desulfurization of liquid fuel has drawn much attention for new regulations requiring (< 10 ppm sulfur) and it is difficult and very costly to use Hydrodesulfurization process for reduction of sulfur content from liquid fuel. In order to find the new regulation, various alternative desulfurization technologies approach such as extractive desulfurization, oxidative desulfurization, desulfurization, adsorption desulfurization and biodesulfurization etc. [10]. Among these desulfurization technologies, oxidative desulfurization (ODS) is the most suitable technique to achieve ultra low sulfur containing compound fuel oils. The main advantage of this process is that it operates at mild operating condition (temperature, Pressure), no need of hydrogen and low operating cost. ODS process has been most attractive method among researchers for removal of refractory sulfur compounds BT, DBT, 4,6-DMDBT etc. [11].

In ODS process various oxidizing agent was used such as ozone, molecular oxygen, hydrogen peroxide (H_2O_2) , organic peracides etc. among these oxidizing agent hydrogen

Figure 2: Oxidative Desulfurization process of sulfur Compound dibenzothiophene (DBT)to sulfone

Jianghua Qiu et al. [13] prepared $H_3PMo_{12}O_{40}/SiO_2$ catalyst for catalytic oxidative desulfurization of fuel oil. The HPMo/SiO₂ catalysts were very efficient for the oxidation of DBT and BT in the model fuel oil by using of H_2O_2 as the oxidant. Under the condition of catalyst dosage 0.05 g, H_2O_2 dosage 0.05 mL and reaction temperature 70°C, the conversion rate of DBT reached 100% only in 60 min and that of BT conversion achieved to 99.2% in 150 min. The kinetic studies indicate that the oxidative desulfurization of DBT and BT was the pseudo-first-order reaction. They also studied the activation energies of BT and DBT was 40.5 kJ/mol and 33.0 kJ/mol, respectively.

N. Jose et al. [14] studied catalytic oxidative desulfurization of the refractory sulfur compound present in the liquid fuel. The investigators used a titanium silicate (TS-1) catalyst for ODS process and sulfur removal achieved to 22% in 240 min by addition of 1.05wt% copper in titanium silicate. They also have done experiments by using the box-behnken method to estimate processes operating condition such as reaction temperature, the catalyst used, and ratio of hydrogen peroxide and thiophene and their values are 70°C, 0.45g, and 19.9 mole respectively to the sulfur reduction achieved to 93%. Yuhua et al. [11] applied catalytic oxidative desulfurization (ODS) for desulfurization of refractory sulfur compounds BT, DBT, 4,6-DMDBT. They also studied that refractory sulfur compound can be oxidized into sulfones by using of hydrogen peroxide oxidant (H₂O₂) over 14wt% MoO_3/γ -Al₂O₃ catalyst under mild operating conditions, sulfur concentration achieved to 100% in 2h. Uttam et al. [16] investigated the oxidation of sulfur compounds in model oil (DBT dissolved in n-octane) by using of hydrogen peroxide (H₂O₂) as an oxidizing agent in the presence of nanocrystal line Ti-beta catalyst. Under mild operating condition such as temperature 100°C, atmospheric pressure, and mole ratio of H₂O₂ to S of 10:1 more than 93.5% of Dibenzothiophene could have oxidized in the model oil. Authors studied the catalytic activity of Ti-beta catalyst, there is significant reduction after the first cycle in DBT conversion.

2.3. Extractive desulfurization (EDS)

Desulfurization via extraction depends on the solubility of the refractory sulfur compound in certain solvents. Babich et al. [17] studies describe general process flow of the

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desulfurization via extraction method as shown in the figure [3]. In the mixing tank feedstock is mixed with the solvent and the refractory sulfur compounds are extracted into solvent because of their higher solubility in the solvent. Then in the separator section, hydrocarbon is separated from the solvent. After the separation, the treated hydrocarbon is blended to the final product or transferred to distillation for further transformation or treatments. During distillation refractory, sulfur compounds are separated from the solvent and the recovered solvent is recycled to mixing tanks. The most attractive characteristic of extractive desulfurization process, it operates at mild operating conditions and no need of hydrogen. The process does not change the chemical structure of fuel components. The efficiency of the extractive desulfurization process is limited by the solubility of the refractory sulfur compounds in the solvent. So a choice of solvent is very important to increase the efficiency of extraction desulfurization process such as solvent should have the different boiling point than sulfur compounds and solvent should be cost effective in order to make feasible for industrial applications. Various solvent has been tried such as acetone, ethanol, and polyethylene glycols, which resulted in 50-90% desulfurization depending on the number of cycles of the process [18].



Figure 3: General process for extractive desulfurization [4]

Various studies have been investigated on the desulfurization via extraction for the removal of the sulfur compound from liquid fuels. Light oils were mixed with different organic solvents such DMSO. acetonitrile as and tetramethylenesulfone at ambient conditions in order to examine he sulfur compounds and aromatic extractability [19]. Topalova et al. studied the two stage desulfurization via extraction with dimethylformamide as a solvent. The result shows that the sulfur content in diesel oil was reduced from 2wt% to 0.33wt% [20]. Xiaochun Chen et al. [21] studied desulfurization of liquid fuels by extraction with Lewis acidic ILs 1-butyl-3-methylimidazolium chloride (ZnCl₂([Bmim]Cl/ZnCl₂). The author also tested the activity of ionic liquid for different desulfurization. The investigators also reported that liquid fuel could be obtained after six stage extraction are obtained at 25°C, ionic liquid/oil ratio of 1, with the yield of thiophene(TS), Dibenzothiophene(DBT) liquid fuels reaches to 93.8% and 95.9% in 30 min. They also investigated the reusability of ionic liquid in desulfurization process.

Swapnil A. Dharaskar et al. [3] reported the extractive desulfurization process of liquid fuels by using of lewisbased ionic liquid ([Bmim]Cl/FeCl₃). They also reported that desulfurization via extraction for removal of sulfur compounds from liquid fuels in single extraction at mass ratio of model liquid fuels to ionic liquid 5:1 at 30°C in the water bath for 30 min and sulfur reduction achieved to 75.6%. They also found that ionic liquid reused without regeneration with the efficiency of 47.3%. Datsevich et al. [22] have studied the profound desulfurization of diesel oil by extraction desulfurization process through ionic liquids. In this study ionic liquid(chloroaluminate) was used and the result showed that by using of five stage extraction process for removal of the sulfur compound from diesel oil achieved to 80% at the temperature of 60° C.

2.4 Adsorptive desulfurization (ADS)

Desulfurization via adsorption is based on the capability of sorbent material to particular adsorb refractory sulfur compounds. Adsorptive desulfurization considered as most economical technology among different desulfurization techniques due to simple operating condition and regenrability of adsorbents such as metal oxides, alumina, metal sulfides, zeolite, silica and activated carbon [23-24]. The adsorptive desulfurization can be approached in two ways: adsorptive desulfurization and reactive adsorption desulfurization. (i)Adsorptive desulfurization occurs through physical adsorption of refractory sulfur compounds on the solid adsorbent surface. Regeneration usually did by thermally spent sorbent with desorbent (ii) reactive adsorption desulfurization based on the interaction of organic sulfur compounds and the adsorbent. Sulfur chemically bounded to the sorbent going in the form of sulfides, as newly formed hydrocarbon compound released into the purified fuel stream. Regeneration of the spent adsorbents results in sulfur elimination in the form of H₂S, S, etc. The Efficiency of adsorptive desulfurization mainly determined by the sorbent material, selectively to refractory sulfur compounds with relative to hydrocarbons, durability and regenerability [17]. The adsorption process is a non-invasive approach which removes sulfur from liquid fuels under mild operating conditions and it in principle has potential for industrial desulfurization. Various research has been conducted to develop materials with high desulfurization capacity or efficiency however even highest efficiency, achieved thus far, is still insufficient for industrial applications. To increase the efficiency of adsorptive desulfurization method the adsorption capacity and sorbent regeneration should be further improved [25].

M.S. Patil et al. [26] reported the desulfurization of hydrocarbons liquid fuels via adsorption. Authors also have done the experiment in a batch reactor for soaking of compounds with sulfur content onto activated carbon, which was furnished with black liquor and phosphoric acid and nitrogen reactor used as the intercalating agent. They also studied, intra particle diffusion resistance has been overcome because of stirring and experimental data obtained by Langmuir adsorption isotherm model. Yoshie shimizu et al. [27] studied desulfurization of fuel oils such as kerosene and diesel oil be adsorption. Authors used rice husks which were carbonized in N₂ at 400°C for 60min and then were activated in CO_2 at 850°C at 60min. the investigators also studied the capacity of rice husk activated carbons to adsorb organic sulfur compounds of Dibenzothiophene were determine by correlating with their textural properties and chemical characteristics. The rice husk activated carbon of 0.5g was soaked in commercial kerosene of 15g at 10°C for 100 h. Jae Hyung Kim et al. [28] have studied adsorptive

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desulfurization and denitrogenation of diesel oil (containing refractory sulfur compounds) over three adsorbents such as activated carbon, activated alumina and nickel based adsorbent in fixed bed reactor. The investigator also studied adsorptive capacity and selectivity for different compounds on the basis of break through curves. The author also reported that activated carbon has high adsorptive capacity and selectivity for both organic sulfur and nitrogen compounds, especially for the organic sulfur compounds with the methyl groups such as 4,6-dimethydibenzothiophene(4,6-DMDBT).

Souman Das Gupta et al. [29] reported adsorptive desulfurization of diesel by used of nickel-based adsorbent. The investigators reported that under optimized conditions (pressure 4 bar and temperature 350°C) nickel based adsorbent(NiMCM-41) could reduce the sulfur concentration from 450ppm to 50ppm. The adsorbent capacity and regenerability were examined by investigators, adsorbent regenerable under controlled oxidation with air at 450°C without loss of the sulfur removal capacity. A.B. Salem et al. [30] studied adsorption desulfurization of naphtha in a batch reactor by using of different solid sorbents such as activated carbon and zeolite 13X. Authors also reported that activated carbon showed high capacity but a low sulfur removal. Zeolite 13X was superior for sulfur removal from low sulfur streams at room temperature. The investigators used two bed combination for the industrial application. The first bed contains activated carbons and capacity to remove sulfur up to 65% at 80°C and the second bed carrying zeolite 13X which is able to remove sulfur content up to 100% at room temperature if the sorbent/feed ratio is bout 800g/l.

Isam A.H. et al. [31] have studied adsorption desulfurization of diesel oil by using of seven different solid sorbents such as Bentonite, Acid activated bentonite, date palm kernel powder, acid activated date palm kernel powder, saw dust powder, commercial powder and granules activated carbon. The experimental study conducted in batch reactor and amount of sorbent used in the range 0-5% by mass at room temperature with contact time 2 h. Authors reported that commercial powder activated carbon got the highest sulfur removal capacity and sulfur content reduced from 410.9 μ g/g to 245.9 μ g/g by using of 5% mass commercial powder activated carbon and 278 μ g/g using acid activated bentonite.

2.5 Biodesulfurization (BDS)

Biodesulfurization (BDS) has been studied an alternative to HDS, BDS is the most efficient process because of it takes place at low temperature and pressure in the presence of microorganism. In the BDS process bacteria used as a catalyst to remove the refractory sulfur compounds from liquid fuels. Caro et al. [32] studied that BDS requires approximately two times less capital cost and 15% less operating cost in comparison with Hydro-desulfurization (HDS) process.

Even So BDS process applying microorganism biocatalyst suitable for desulfurizing of organic sulfur compound such alkylated DBT is good for this purpose and various studied had done by researchers on BT, DBT desulfurizing bacteria and their enzyme involved in removal of sulfur compounds from fuel. The life of microorganism in BDS process used to be short (1-2 days), but this has been extended to 8-16 days. However, current design allows the production and regeneration of the biocatalyst within the BDS process which provides 200-400 h biocatalyst life. [17]. The biodesulfurization process carried out in two phase system, whether whole cell used as biocatalyst in the aqueous phase interacts with oil phase [4,33]. Long ago only gram positive bacteria utilized for biodesulfurization of sulfur compounds.

Gunam et al. [34] applied gram bacteria (Sphingomonas subarctica T7b) for degradation of sulfur compounds (DBT, 4,6-dibutyl DBT and 4,6-Dipentyl BDT) from liquid fuels. They also studied the ability of Sphingomonas subarctica T7b by using of resting and immobilized cells with DBT, alkyl DBT and commercial light gas oil as the substrate. The resting cells of Sphingomonas subarctica T7b degraded the DBT from 250 to 239.2 mg/l within 24 h at 27°C, while 127.5 mg of 2-hydroxybiphenyl (2-HBP) was formed in the result 55% DBT conversion achieved [30]. The investigators also investigate that cells immobilized by entrapment with polyvinyl chloride exhibit high desulfurization activity of DBT and it could be used more than 8 cycles. The stability of immobilized cells was better than that of resting cells at different pHs, temperature, and DBT desulfurization. The desulfurization process carried out in two pathways: aerobic pathways (kodama pathways) and 4S pathways (sulfur specific pathways) but most attention given to 4S route for removal of refractory sulfur compounds from fuels. The pathway of 4S include four consecutive reactions as shown in the Fig: 4 [32]. In beginning DBT is oxidized by two monooxygenases, DBT monooxygenase(Dszc), DBT sulfone monooxygenase (Dsza) and at the end converted to 2hydroxybiphenyl by the desulfinase(DszB).

Aribike et al. [35] reported the biodesulfurization(BDS) of diesel fuel by desulfobacterium anilini. The investigators also studied the used of desulfobacterium aniline isolated from petroleum products- polluted soil and the removal of sulfur containing hydrocarbons from diesel oil. Authors carried out experiments at 300°C and atmospheric pressure and concluded that the peaks of benzothiophene(BT), dibenzothiophene(DBT) in diesel oil decreases after applied of BDS process. Author reported that at the end of 72 h yield of diesel oil reached to 82%. A newly identified bacterial strain Rhodococcus sp.(JUBT1) isolated from diesel oil, which has been utilized for different refractory sulfur compounds such as, DBT, alkylated DBT etc. and which basically not changed during hyodrodesulfurization of diesel oil. the desulfurization via microorganism of refractory sulfur compounds achieve to 1000-100 mg/dm³ in 24h [36].

Figure 4: The 4S route for the biodesulfurization of DBT and its derivatives [17].

Figure 5: Biodesulfurization through Kodama pathways for DBT (a) dibenzothiophene; (b) cis-1,2-dihydroxy-1,2dihydrodibenzothiophene; (c) 1,2-dihydroxydibenzo thiophene; (d) cis-4- [2-(3 hydroxy) thioaphthenyl]-2-oxo-3butenoate; (e) trans-4-[2-(3-hydroxy) thioaphthenyl]-2-oxo-3-butenoate; (f) 3-hydrooxy-2-formylbenzothiophene.

Fatemeh et al. [33] applied *Rhodococcus erythropolis* (SHT87) strain which was isolated from oil contaminated soils in Iran for biodesulfurization of organosulfur compounds. *Rhodococcus erythropolis*(SHT87) was found to three sulfur metabolizing genes such as DszA, DszB, DszC, which indicates that SHT87 strain converts dibenzothiophene to 2-hydroxybiphenyl(2-HBP) through 4S pathway. Author studied growth of this strain in 5g/L glycerol as carbon source in 120 h by cultivation of organism in 120 min, conversion was achieved to 30% which derived from the

equal amount of 2-hydroxybiphenyl(HBP). Metabolizing enzymes having the maximum specific activity in the range 0.36-0.47 μ mol HBP min⁻¹. Author also studied bacterial growth by using of haladane type kinetics characterizing the presence of substrate inhibition.

Gupta et al. [38] reported the biodesulfurization of sulfur containing compounds such as DBT and its derivatives via kodama pathways. In this route or pathways initial oxidative C-C cleavage, which involves ring cleavage of one aromatic ring in sulfur compounds as shown in the Fig: 5.

3. Conclusion

Different technologies were proposed for the desulfurization of liquid fuel. These technologies include Hydrodesulfurization, extractive desulfurization, oxidative desulfurization and adsorptive desulfurization. Among these desulfurization technologies few of the strategies are viable for the deep desulfurization of liquid fuels. Because of properties of liquid fuels like, high boiling point, more refractory sulfur content and the nature of refractory sulfur compounds.

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