

Catalytic Oxidative desulfurization of liquid fuels: A review

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Abstract - Oil desulfurization technique can be broadly classified into two categories: Hydrodesulfurization (HDS) and non-HDS. Among the various non-HDS technique available, the oxidative desulfurization (ODS) have been most effective for the removal of sulfur from fuel oil. In these review, we had systematically analyze the various oxidative system like Formic acid/ H₂O₂ oxidation system, Heteropolyacid (HPA)/ H₂O₂ oxidation system, Ionic liquid/ H₂O₂ oxidative system, Molecular sieve/ H₂O₂ oxidation system, Poloxometalates/ H₂O₂ oxidation system, TS-1/ H₂O₂oxidation system, Ultrasound/ H₂O₂ oxidation system frequently used for ODS technique.

Key Words: Hydrodesulfurization, Non HDS, Oxidative desulfurization, Oxidative System etc.

1. INTRODUCTION

Desulfurization of crude oil is one of the important processes in the refining of crude oil to final product. In recent times, the required specification for transportation fuels have become increasingly stringent with respect to sulfur content. Efforts are being made to produce petrochemical products and transportation fuels to be almost sulfur free. Consequently, the removal of sulfur from the crude oil has become one of the prime processes and consequently, the price of a crude oil is influenced by its sulfur content [1].

The stringent environmental regulation has limited the level of sulfur content in many countries. The limit have been less than 15 µg/g since 2006 in US, less than 10 µg/g in Europe since 2005, and less than 50 µg/g in Beijing and shanghai in china since 2008 [2]. The new regulation in India since April 1, 2010 has prescribed the sulfur content limit for gasoline and diesel to be 50 µg/g with the limit reducing to as low as 10 µg/g in near future [3]. The sulfur content can poison the noble metal catalyst used in the reforming and transforming process of fuel and the electrode catalysts used in fuel cell stacks [2]. Due to this stringent condition, the refineries are facing the major problem in treating the sulfur content crude oil and the need for ultra deep desulfurization of liquid

hydrocarbon fuels has become an increasingly important subject worldwide.

The main traditional and widely used method available is hydrodesulfurization which employ the use of hydrogen at an elevated pressure (20-100atm of H₂) and high operating temperature of 300-400 °C . The method is highly effective for aliphatic and acyclic S-compound but less effective for the treatment of aromatic sulfur compound. However, the existing hydrodesulphurization technique require much harsher operating condition to remove the last 100 ppm of sulfur due to the presence of refractory sulfur compound such as benzothiophenes (BT) and dibenzothiophene (DBT), which demand higher hydrogen pressure, temperature and /or contact time to achieve a fuel with sulfur concentration of less than 10 ppm [4,10-11], ultimately increasing the initial and operating investment cost. Therefore there is a need to develop alternative cost efficient method which operates at normal range temperature and pressure for ultra-deep desulfurization process. Many efforts have been made in these direction and new methods such as reactive adsorption or adsorptive desulfurization [5-9], extractive distillation [6], oxidative desulfurization [7], biodesulfurization [8] has gained much recognition in recent times.

Oxidative desulfurization (ODS) is the most interesting desulfurization technique among the researcher. Many research are being carried on this subject. ODS process primary include two stages: (i) Oxidation of sulfur compound and (ii) Liquid extraction. The process makes use of oxidant to oxidize the organic sulfur present in fuel oil and is subsequently removed by adsorption or extraction. The primary focus of this review is to summarize the various oxidative system used in ODS. The sulfur compound like benzothiophene, dibenzothiophene, 4,6- Dimethyldibenzothiophene (4,6-DMDBT) are oxidized using selective oxidant to a compound that can be preferentially extracted using suitable solvent. The various oxidative system such as Formic acid/ H₂O₂, Ionic liquid/H₂O₂, Heteropolyacid (HPA)/ H₂O₂, Molecular sieve/ H₂O₂, Poloxometalates/H₂O₂, TS-1/H₂O₂, Ultrasound/H₂O₂ is deeply analyzed based upon the literature survey in the present review work.

2. CATALYTIC OXIDATIVE SYSTEM

2.1 Formic acid/ H₂O₂ oxidation system:

The reactivity of sulfur compounds for oxidation increases with increase of electron density on sulfur atom. The reactivity of refractory sulfur compounds decreases in order of 4,6-DMDBT > 4-MDBT > DBT. The order of reactivity in HDS of sulfur compounds is reverse of the above pattern.

Yao Xiuqing et al. [12] studied the desulfurization process for DBT and its derivatives such as MDBT, 4,6-DMDBT by using formic acid as catalyst and H₂O₂ as an oxidant. Model sulfur compounds of BT, DBT and 4,6-DMDBT is dissolved in n-heptane to prepare the model oil. The results shows that under the condition of the reaction temperature of 60°C, the molar ratio of hydrogen peroxide to sulfur of 7:1, and volume ratio of hydrogen peroxide to formic acid of 1:1, and the reaction time 40min. The conversion achieved for 4,6-DMDBT, DBT and BT were 100%, 96% and 58% respectively.

Alireza Haghghat mamaghani et al. [14] studied the oxidative desulfurization of model fuel by using of formic acid as a catalyst and H₂O₂ as an oxidant. For deep desulfurization of sulfur containing compounds (BT, DBT, 4,6-DMDBT) sulfuric acid was used to increase the acidity of model sulfur compounds and catalytic activity with formic acid. The sulfur containing compounds is oxidized to sulfones followed by liquid-liquid extraction using acetonitrile to remove sulfones from model oil. The result shows that under operating condition, reaction temperature of 65°C, molar ratio of H₂O₂ to sulfur of 2:1, ratio of formic acid to sulfur of 22:1 and reaction time of 56min, 100% conversion is achieved.

H₂O₂-organic acid oxidative system has relatively mild reaction conditions and strong oxidizing ability, to achieve high desulfurization rate, but the organic acids in the oil-soluble part impact the fuel quality. And the liquid organic acids have several disadvantages such as unrenewable and a high reclaiming cost, so catalyst which can be recovery is one indispensable choice [13].

2.2 Heteropolyacid (HPA)/H₂O₂ oxidation system:

Heteropolyacid has various advantages such as (i) readily available (ii) Bronsted acidity (iii) easy to handle (iv) nontoxic. Researchers widely used HPA on molecular level. From last decades HPA was used in oxidative desulfurization process and H₂O₂ used as an oxidant [15]. **heteropoly** compounds easily mixed and the capability for introduce elements into their structure that required to develop the desired properties [16].

Silva Marcio Jose da Silva et al. [17] applied keggin Heteropolyacid (HPA) with alumina catalyst for the oxidative desulfurization of model oil. Model oil was

prepared by dissolution of model sulfur compounds (BT, DBT) into isooctane and H₂O₂ is used as an oxidizing agent because it is environmental friendly. Model oil was oxidize into sulfones and extracted by acetonitrile. The sulfur reduction achieved was 1000ppm to less than 1ppm in 180min at 60°C.

Ling tang et al. [18] applied Heteropolyacid (HPA) catalyst with keggin structure such as H₃PW₁₂O₄₀ (HPW) for oxidative desulfurization of model oil and H₂O₂ used as oxidant. The catalyst has high catalytic activity for the desulfurization of refractory sulfur compounds such as Benzothiophene (BT), Dibenzothiophene (DBT). They also modified the HPA catalyst with quaternary ammonium salt. The reaction occurs at very mild operating condition and sulfur conversion achieved to 98.1% at 60°C in 180min. the researchers found that after three recycled of the catalyst no loss observed in catalytic activity.

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2.3 Ionic liquid/H₂O₂ oxidation system:

Wei Jang et al. [19] investigate ionic liquid dialkylpiperidinium tetrachloroferrates such as [C₂OHmpip]FeCl₄, [C₄mpip]FeCl₄, [C₈mpip]FeCl₄ and [C₁₂mpip]FeCl₄ catalyst for oxidative desulfurization of model oil. The solution of refractory sulfur compounds such

as Dibenzothiophene(DBT) dissolved in n-octane were used as model oil. The result showed that conversion achieved to 97.1% of Dibenzothiophene(DBT) by using of catalyst $[C_4mpip]FeCl_4$ in ionic liquid $[C_8mim]BF_4$ at 303K in 1hr and molar ration of H_2O_2/S was 3.5:1. They reported that catalyst very efficient because it could be recycled four times without significant decrease in catalytic activity. The catalytic activity of refractory sulfur compounds decreased in order of $DBT > BT > 4,6-DMDBT$.

Fa Tang Li et al. [20] take a kind of ionic liquid catalyst ($C_5H_9NO.SnCl_2$) which was prepared by reaction of N-Methyl Pyrrolidone with anhydrous $SnCl_2$ for oxidative desulfurization of Model oil and H_2O_2 used as oxidant. Model oil prepared by Dibenzothiophene(DBT) dissolved in n-octane. Desulfurization of Dibenzothiophene(DBT) in oxidative desulfurization by using of ionic liquid yield achieved to 94.8% at 30°C in 30min. in the operating condition of molar ratio of H_2O_2 to DBT is 6 and volume ratio of ionic liquid to model oil is 1:3. They also studied activation energy of the catalyst was 32.5 kJ/mol. The ionic liquid catalyst could be recycled 6 times without any loss in catalytic activity.

2.4 Molecular sieve/ H_2O_2 oxidation system:

Molecular sieves mostly used in oxidative desulfurization process(ODS) for alkylation of aromatics olefins ethylene and oxidation of reaction[21]. Molecular sieve has high catalytic activity for desulfurization due to large pore size [26].

Lanju, Chen., Guo, Shaohui[22] applied formic acid oxidative system over cerium oxide loaded molecular sieve in the solution of model sulfur compounds of thiophene(C_4H_4S) and 3-methylthiophene(3- MC_4H_4S) in n-heptane and H_2O_2 used as oxidant. The cerium oxide-loaded molecular sieve was very active catalyst for the oxidation of thiophene (C_4H_4S) and 3-methylthiophene (3- MC_4H_4S), with 78.4% and 82.3% sulfur removal rate respectively. Phase transfer catalyst improved the sulfur removal rate of C_4H_4S and 3- MC_4H_4S in the oxidation reaction system. However, the sulfur removal rate of C_4H_4S and 3- MC_4H_4S was decrease with the addition of cyclohexene and xylene into the solvent n-heptane.

Cheng, Shifu, et al. [23] applied Ti-MWW catalyst in oxidative desulfurization of the model light oil and oxidizing agent used as H_2O_2 . Sulfur containing compounds such as benzothiophene(BT), dibenzothiophene(DBT) first oxidize into sulfoxide and sulfones and after that extracted by acetonitrile. The conversion of benzothiophene(BT) and dibenzothiophene(DBT) at 70°C achieved 100% and 95% respectively.

Changi Jin et al. [24] investigated an oxidative desulfurization process for model oil over molecular sieve catalyst Ti-HMS/TS-1. Sulfur containing compounds such as benzothiophene(BT) and dibenzothiophene(DBT) are oxidized to convert in sulfones and sulfoxides respectively and H_2O_2 used as oxidant because it has several advantages

such as low cost and environment friendly [25]. Ti-HMS is Ti containing molecular sieve catalyst which holds large pores(2-3nm). The conversion rate of DBT and BT is 100% and 98% respectively. They also studied oxidative desulfurization test of thiophene was less as compared to BT and DBT due to lower oxidative ability holds low catalytic activity.

1.5 Poloxometalates/ H_2O_2 oxidationsystem:

One of the catalyst investigated by some researchers which is solid acid oxidation catalyst it has large molecular size, good transmission and storage capacity to electrons and protons, good thermal stability. Tungstophosphoric acid(TPA) is kind of heteropoloxometalates catalyst used in oxidative desulfurization[27]. Tungstophosphoric acid (TPA) catalyst in acetic acid and reaction done with the solution of organic sulfur compounds such as dibenzothiophene(DBT) and benzothiophene(BT) in Octane and H_2O_2 used as oxidant. They found that the oxidation reaction happened in biphasic system. The oxidation reaction takes place in AcOH phase, and most of the oxidation products remain in this phase and it occurs successive removal of sulfur compounds from the octane phase at very low temperature.

Gwang-Nam Yun et al. [28] applied Phosphotungstic acid for oxidative desulfurization of light cycle oil(LCO) and H_2O_2 used as oxidizing agent. Refractory sulfur compounds oxidized by hydrogen peroxide to convert in sulfones and sulfoxides and solvent extraction used to remove sulfur from sulfones product. the reactivity of refractory sulfur compounds in ODS process was $DBT > 4-MDT > 4,6-DMDBT > BT$ due to electron density on sulfur atoms and structural properties of refractory sulfur compounds. The results showed that nitrogen compounds reduce the overall ODS activity but present of aromatic compounds recovered the ODS catalytic activity.

C. Momintarachat et al. [29] studied the oxidative desulfurization over polyoxometalates catalyst for removal of sulfur content from model oil (model oil is the solution of refractory sulfur compounds such as BT, DBT, 4,6-DMDBT and n-Octane). Refractory sulfur compounds oxidized in sulfones by using of H_2O_2 /Acetic acid. After that they used solvent extraction and absorption process to remove sulfur from sulfones product. The sulfur removal achieved to 99% at very mild operating condition. The results showed that catalyst has high catalytic activity.

1.6 TS-1/ H_2O_2 oxidation system:

In recent time TS-1(Titanium silicate) is widely used in oxidative desulfurization process for removal of sulfur from refractory sulfur compounds. Researchers studied catalytic activity of TS-1 catalyst over refractory sulfur compounds under mild operating conditions [30-31].

N. Jose, S. Sengupta, et al. [32] investigated an oxidative desulfurization process for model oil over copper loaded titanium silicate (TS-1). Refractory sulfur compounds dissolved in iso-octane to form model oil. Refractory sulfur compounds such as thiophene, benzothiophene and dibenzothiophene oxidized to sulfones and sulfoxides by using of oxidizing agent H_2O_2 . They also use Box-behnken method to find out the operating condition and they found the temperature of reaction to be $70^\circ C$, catalyst amount 0.45g, and molar ratio of H_2O_2 to thiophene is 19.9 to achieve a conversion of 93%. The activation energy of catalyst was found to be 18.76 kJ/mol.

C. Shen, et al. [33], applied porous glass beads supported on TS-1 catalyst for oxidative desulfurization of model fuel. The model fuel is prepared by refractory sulfur compounds such as dibenzothiophene (DBT), 4,6-dimethyldibenzothiophene (4,6-DMDBT) dissolved in n-octane. DBT and 4,6-DMDBT oxidized to sulfones or sulfoxide by using H_2O_2 as oxidizing agent. The conversion of dibenzothiophene and 4,6-dimethyldibenzothiophene achieved is 100% and 56.3% respectively at $70^\circ C$ in 2 min by using the catalyst with Ti amount 0.507wt%. The used catalyst can be regenerated by heating at high temperature and it could be recycled without any decrease in catalytic activity.

1.7 Ultrasound / H_2O_2 oxidation system:

Ultrasound oxidation system is a new oxidative desulfurization technique for deep desulfurization. In ultrasound oxidation system sound energy is used for desulfurization. Ultrasound desulfurization occurs in following path such as mixing of raw material and oxidant with surfactant in water in reactor to prepare a mixture of water and organic phases [35]. Ultrasound oxidative desulfurization process is the combination of three major process (i) Phase transfer catalysis (ii) Transition metal catalysis and (iii) mild operating condition [36].

Fabio A. Duarte et al. [34] (2011) studied on desulfurization process of model sulfur compounds such as Benzothiophene (BT), Dibenzothiophene (DBT) and 4,6-Dimethylbenzothiophene (4,6-DMDBT) by combination of photochemical reaction and liquid-liquid extraction. At optimum condition the sulfur conversion from model sulfur compounds achieved is 99% at $90^\circ C$ in 9 min by using molar proportion for H_2O_2 : acetic acid: sulfur of 64:300:1. The author also find out that sulfur removal without photochemical oxidation desulfurization was lower than 82% for model compounds and 55% for diesel. Catalytic process need less amount of H_2O_2 .

Hai Mei et al. [37] applied ultrasound oxidative desulfurization to remove sulfur from model oil. Model oil is the mixture of refractory sulfur compounds such as Thiophene Benzothiophene (BT), Dibenzothiophene (DBT), 4,6-Dimethyldibenzothiophene (4,6-DMDBT) in n-octane. Refractory sulfur compounds oxidized to form thiophene sulfones, Dibenzothiophene sulfones and 4,6 dimethyldibenzothiophene sulfones and remove sulfur from

model oil by applying ultrasonic energy and solvent extraction. They found that sulfur conversion achieve is 99% in short period of time at very mild operating condition.

2. CONCLUSIONS

Though HDS process has been the dominant technology for the desulfurization process, it suffered from major drawback of high investment, severely harsh operating condition and unable to meet increasingly stringent environmental regulation of ultra low sulfur fuel oil. The stringent environmental regulation demands the development of non-HDS technique for the ultra low sulfur fuel oil. The ODS technique has a huge potential and presently it has shown some remarkable achievement in the desulfurization technique, a number of problem has still to be figured out such as high concentration of oxidants, catalyst deactivation, high cost involved in solvent extraction, low efficiency of oil removal. The various aspect of work that is proposed to address the problem faced in ODS technique and to achieve practical application are; development of low cost and high efficiency recoverable catalyst, development of low cost oxidant, actively search and carefully assessing the new desulfurization technique such as photochemical oxidation and plasma oxidation to promote the growth of ODS technique so as to fulfill the requirement of society.

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